

# **ADDITIVE EFFECTIVENESS INVESTIGATIONS IN ALTERNATIVE FUELS**

**INTERIM REPORT  
TFLRF No. 448**

**by  
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Principal Scientist**

**U.S. Army TARDEC Fuels and Lubricants Research Facility  
Southwest Research Institute® (SwRI®)  
San Antonio, TX**

**for  
Allen S. Comfort  
U.S. Army TARDEC  
Force Projection Technologies  
Warren, Michigan**

**Contract No. W56HZV-09-C-0100 (WD17–Task 9)**

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U.S. Army TARDEC Fuels and Lubricants  
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## EXECUTIVE SUMMARY

This program examined the effect of long term, accelerated storage (36 weeks at 43 °C) on thirty (30) alternative fuel samples. The base materials for these samples were three alternative fuel products: FT SPK and HEFA SPK representing current aviation turbine fuel alternative materials and B20 representing the highest percentage use of alternates in distillate fuels. The samples were further subdivided by the programmatic addition of one biocide and three cetane improvers. In addition, the turbine fuel materials were also treated with the standard JP-8 additive package. Finally, after the additive addition, the samples were inoculated with a live culture to assess the biologic potential of these alternative fuel materials.

The samples were tested, in accordance to the relevant standard, at the start and at the end of the storage period. This testing was limited to the standard specification properties for the class of fuel being tested. The testing did not include the more rigorous investigation of fuel properties for a full fit-for-purpose evaluation but it was the appropriate first step in any such evaluation.

There was a lot revealed by this program but it may well be surmised by two major insights:

- 1) System cleanliness is paramount in preventing biologic activity
- 2) Additive qualification is a complex task that may well require more effort than simply verifying the product performs the stated function.

The biologic concern for the materials in this program was that they represented near ideal foods for microbial infestations. The program generated a successful live culture and introduced it to the storage samples with these ideal foods and sufficient water yet, essentially, nothing happened. The working theory is that the system simply did not have enough nutrients (typically materials that provide the elements needed for life, such as nitrogen and phosphorous) to sustain growth. This clearly points to the need to need to maintain clean, dry fuel supplies.

Even when performance additives provide the stated benefit, simply judging the product on that success is not sufficient. For instance, in this program one of the cetane improvers promised both

improved cetane value and storage stability for biodiesel. If only those properties were measured, it would have been deemed a success but a broader study covering both diesel and turbine fuel showed it to be a very poor candidate for use in military systems where cross contamination, due to dual use, is a real potential. Conversely, the testing showed that, in a clean system, standard cetane improvers may well be more stable than expected.

In terms of material compatibility, the current aviation industry practice for additive approval only requires that the additive be shown not to affect the basic properties of the fuel. The inter-additive evaluation is limited to assessing the solubility stability of candidate additives. In this program the various additive combinations were subjected to the full specification testing and this effort demonstrated there can be negative synergistic effects.

The overall program generated a dozen key points that were further consolidated into five major conclusions on the potential impact of the information generated on the use of these fuels and additives. This, in turn, led to the generation of twelve recommendations on how to use the information to improve future implementation of alternative fuels and new additives.

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The authors would like to acknowledge the contribution of the TFLRF technical and administrative support staff.



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## ACRONYMS AND ABBREVIATIONS

2EHN	2 Ethyl Hexyl Nitrate, cetane improver
ASTM	ASTM International, consensus standard organization
ATP	Adenosine TriPhosphate
B20	Biodiesel, 20% blend with refined diesel (min)
BOCLE	Ball on Cylinder Lubricity Evaluation, ASTM D5001
CFPP	Cold Filter Plugging Point, ASTM D6371
CI	Compression Ignition
CI/LI	Corrosion Inhibitor / Lubricity Improver
CLxx-xxxx	SwRI Sample ID, Chemical Lab - Year (xx) - Sequence Number (xxxx)
CN #1	Cetane Improver #1 – 2 ethyl hexyl nitrate (2EHN)
CN #2	Cetane Improver #2 – 2EHN, with storage stability additives
CN #3	Cetane Improver #3 – Di Tertiary Butyl Peroxide (DTBP)
CONUS	Continental United States
DCN	Derived Cetane Number by IQT
DiEGME	Di Ethylene Glycol Methyl Ether
DLA-Energy	Defense Logistics Agency, Energy Group
DTBP	Di Tertiary Butyl Peroxide, Cetane Improver
EOT	End of the Test Program
EPA	Environmental Protection Agency
EU	European Union
FAME	Fatty Acid Methyl Ester, aka Biodiesel
FOG	Waste Fat, Oil and Grease from Food Processing and Preparation
FSII	Fuel System Icing Inhibitor
FT SPK	Synthetic Paraffinic Kerosene from the Fischer Tropsch Process
HEFA SPK	Synthetic Paraffinic Kerosene from Hydroprocessed Esters and Fatty Acids
HFRR	High Frequency Reciprocating Rig, ASTM D6079
HRJ	Generic term for bio derived jet fuel
IQT	Ignition Quality Test, ASTM D6890
JFTOT	Jet Fuel Thermal Oxidation Test, ASTM D3241
MSEP	Micro Separatometer, ASTM D3948
NRL	Naval Research Laboratory
RLU	Relative Light Units
SDA	Static Dissipater Additive
SOP	Standard Operating Procedures
SOT	Start of the Test Program
SwRI	Southwest Research Institute
T18	Midterm, eighteen weeks into the program
TFLRF	US Army TARDEC Fuels and Lubricants Research Facility (SwRI)
UK(MOD)	United Kingdom, Ministry of Defence
ULSD	Ultra Low Sulfur Diesel
US	United States

## 1.0 INTRODUCTION

### 1.1 BACKGROUND

The U.S. Army investigated the property effects generated by the addition and long term storage of additives. The additives tested were one type of biocide and three types of cetane improvers. Since there was a long history of using these materials in standard refined fuels, the emphasis was on alternative fuels. To that end, the program was designed to look at biodiesel and synthetic paraffinic kerosene. Because there is a generally held belief that these materials are much more susceptible to a biologic attack, another key point of the program was a biologic challenge.

### 1.2 PROGRAM

The program, based on the statement of work for Task 9 of WD 017 Revision 002, is detailed in Appendix A. In brief, the project consisted of preparing thirty samples, as identified in Table 1. These samples were tested for specification properties upon blending and then at the end of a 36 week storage at 43 °C. Before entering storage, the samples were dosed with a live culture and water.

**Table 1. Program Test Matrix**

	<b>B20 Baseline</b>	<b>FT-SPK Additized per MIL-DTL-83133 G Baseline</b>	<b>HEFA-SPK Additized per MIL-DTL-83133 G Baseline</b>
No Additional Additives	X	X	X
Biocide	X	X	X
CN Improver #1			
max treat rate	X	X	X
1/2 max treat rate		X	X
CN Improver #2			
max treat rate	X	X	X
1/2 max treat rate		X	X
CN Improver #3			
max treat rate	X	X	X
1/2 max treat rate		X	X
Biocide + CN Improver #1			
max treat rate	X	X	X
Biocide + CN Improver #2			
max treat rate	X	X	X
Biocide + CN Improver #3			
max treat rate	X	X	X

## **2.0 PROGRAM MATERIALS**

### **2.1 TEST FUELS**

Historically, the U.S. Army has relied on a mix of kerosene and distillate range petroleum fuels. In the last decade there has been a drive to introduce alternative materials into the fuel supply. The primary alternative material in the distillate range is Fatty Acid Methyl Ester (FAME, aka Biodiesel). Alternatives for the kerosene range are primarily designed for use in jet fuel and, thus, are hydrocarbons.

#### **2.1.1 Synthetic Paraffinic Kerosene**

The aviation fuel industry has been moving steadily to include fuel components from alternative sources. Where there had been significant resistance to alternative fuels in the automotive sector, the aviation industry said yes, but due to the unique operational issues involving jet fuel any alternative fuel source has to be a hydrocarbon.

Starting in the 1990s, with the efforts by SASOL to get their IPK approved for use in UK(MOD) DS91-91 [1], through the present where two types of synthetic paraffinic kerosene (SPK) are approved for use in both military specifications, MIL-DTL-83133H [2], and commercial specifications, UK(MOD) DS91-91 and ASTM D1655 [3] (via ASTM D7566 [4]). As a production item, SPKs are tested to a narrow set of criteria designed to prove their purity. In this program, however, they were subjected to the normal requirements for a fully formulated jet fuel. In this case, those listed in MIL-DTL-83133G (current at the start of this program).

#### **FT SPK**

FT SPK refers to synthetic paraffinic kerosene generated through the Fischer-Tropsch process. Basically, a carbon source (coal, natural gas, biomass) is converted to process gas (carbon monoxide, CO, and hydrogen, H) and then catalytically reassembled into larger hydrocarbons. There are two basic types; iron catalyzed (as produced by SASOL in South Africa) which produces a highly branched paraffin and cobalt catalyzed (all others to date) which produces a *n*-paraffin (linear) wax.

The highly branched paraffin would not be expected to support much biological activity, so the program selected a cobalt catalyzed product. Because they are not suitable, property wise, as generated, the normal paraffins of the cobalt process are then subjected to hydroprocessing and catalytic treatments to produce a minimally branched (methyl and dimethyl alkanes) paraffin. This still results in relatively long chains of normal paraffins that would be suitable for biologic activity.

A detailed discussion of FT SPK can be found in the Coordinating Research Council (CRC) Report [5] that was used for justifying, including FT SPKs, in the specifications. There were four cobalt FT process SPKs in that report, two from SASOL and one each from Shell and Syntroleum. SwRI had a good supply of the Shell FT SPK and that material was used for this project. This fuel has been in storage for several years and, as a result, provided an ‘aged’ sample which covers another factor against which to evaluate the additive effects. (The effect of aging can be seen in the existent gum, ASTM D381, results for the FT SPK blends in Table C-5.

### **HEFA SPK**

HEFA SPK refers to the synthetic paraffinic kerosene generated by hydroprocessing esters and fatty acids (from triglycerides). The first step is to use hydrotreating to remove the oxygen from the esters and triglycerides and produce a paraffin wax. The resultant wax is then subjected to the same type of hydroprocessing that is used with FT derived paraffin wax and, thus, generates a very similar, minimally branched paraffin.

A detailed discussion of HEFA SPK can be found in ASTM Research Report RR:D02-1739 [6]. That report included detailed information on HEFA SPKs processed from a wide variety of sources. One of those products was Syntroleum R8, produced from waste fats, oils and greases (FOG). There was sufficient R8 on hand at SwRI to provide the needed material for this program. It is generally considered that FOG is the biggest challenge from which to make a satisfactory final product so this choice added another clear factor against which to consider the results.

### **2.1.2 FAME (Biodiesel)**

FAME is made by transesterifying organic fats and oils. The process mixes methanol with the fat and oil in the presence of a catalyst. The catalyst acts to exchange the methanol into the carboxylate bond and release the glycerin backbone. The neat product, B100 (for 100% Biodiesel), is described in ASTM D6751 [7]. The material is primarily used in blends with petroleum products, and that is designated by combining B, for biodiesel, with the desired percentage (Example B5 is 5% biodiesel).

Common biodiesel dilutions are B5 (US) and B7 (EU) for general commercial biodiesel and B20 (US) and B30 (EU) for limited fleet applications. The latter category covers a significant amount of biodiesel acquired by DLA-Energy, the fuel acquisition arm of the U.S. Defense Logistics Agency, in accordance with U.S. environmental policy. It is generally assumed that the higher the biodiesel percentage the more the blend will be susceptible to biologic contamination. Based on that, the intent was to use a higher biodiesel blend.

### **B20**

While B20 is a standard grade of biodiesel, it is not widely available. When unable to locate a ready supply, the program blended B100 with ultra low sulfur diesel (ULSD). While the initial testing indicated the blend was correct, testing the final blends proved the content was high, about 23.8% (See Table C-5 ). Since the driving point of the project was to investigate the issues associated with alternative fuels, and the value is bracketed by U.S. and EU specifications, this was seen as minor issue. The biodiesel blends were tested in accordance with ASTM D7467 [8].

## **2.2 TEST ADDITIVES**

The U.S. Army TARDEC Fuels and Lubricants Research Facility at SwRI (TFLRF) routinely tests commercial and military jet fuel, diesel fuel and variations thereof. Additionally TFLRF routinely works with the standard additives associated with these fuels. As a result, TFLRF is used to working with these materials in a safe fashion. This program dealt with additive materials

not commonly used in these facilities. This required, per SwRI SOPs, a review of the health and safety issues surrounding these materials. Based on that review a chemical hygiene plan was prepared and is included in Appendix B.

The hazards, such as toxicity and explosive decomposition, discussed in the chemical hygiene plan suggest due care before introducing these materials into general use as approved additives. Any perceived benefit of their use could be negated by the potential health and equipment damage issues.

### **2.2.1 Biocide**

The only approved biocide for use diesel fuel acquired by the U.S. Army is Kathon™ FP 1.5 Biocide. While MIL-DTL-83133H does not specifically identify biocides, this material is approved for the commercial jet fuel specification, ASTM D1655. Unlike other additives, such as corrosion inhibitor / lubricity improver (CI/LI), Kathon is not approved for continuous or unrestricted use (see Table 2 of ASTM D1655). In fact, the IATA (International Air Transport Association) guidance material [9] warns that continuous use could result in an incurable biomass accumulation.

The active components of this product are a mixture of methyl and chloromethyl isothiazolinone. At high concentrations, such as the 1.3-1.7% in this product, it requires careful handling. At low concentrations, < 20 mg/kg, these materials are routinely used in cosmetics. For fuel use, the supplier recommended a treatment rate of 100 ppm, based on a volume/volume dilution of 1 part of Kathon to 10,000 parts of fuel. Thus, at the application rate the material is probably not a significant health hazard.

### **2.2.2 Cetane Improver**

Cetane is the common name for *n*-hexadecane, a C<sub>16</sub> normal paraffin. This material is considered to represent the maximum ignition value for a compression ignition (CI) engine fuel. It is used to calibrate the high value, 100 Cetane, of a single cylinder CI test engine. The low value is set



at 15 Cetane using the isoparaffin, hepta-methyl-nonnane (a C<sub>9</sub> alkane with seven methyls, C<sub>1</sub>, attached). The Cetane Number of a test fuel is determined by comparing its test CI performance against specific ratio admixtures of the calibration fluids, as described in ASTM D613 [10].

Typical CI engines are designed to work best with fuels having a Cetane Number range of 40 to 60. If the U.S. Army only used diesel fuel, this would not be an issue. The problem is that they are obligated to comply with the Single Fuel Forward doctrine and, thus, they rely primarily on turbine fuel in the field. Turbine fuel is, by nature, a very low temperature material with a maximum freeze point, for JP-8, of -47 °C. Getting such a low freeze point requires reducing the normal paraffin content of the fuel, thereby increasing the ratio of isoparaffins. As discussed in the previous paragraph, that drives down the Cetane Number of the jet fuel. Substantial amounts of jet fuel, greater than 13% of JP-8 according to a recent DLA survey [11] for instance, have Cetane Number values below 40.

There are other chemistry issues, such as aromatics and cycloparaffins, that can result in a lower Cetane Number, so the petroleum industry has developed materials that will help promote ignition. These are referred to as cetane improvers. There are two primary types of cetane improver:

- 1) Octyl Nitrate – The most common version being, 2-ethyl-hexyl-nitrate (2EHN).
- 2) Di-Tertiary-Butyl-Peroxide (DTPB) – This is primarily used in areas where nitrous oxide is a particular issue.

For this project SwRI acquired three versions of cetane improver:

- 1) 2EHN (CN #1), with a maximum treatment rate of 0.80 vol%.
- 2) 2EHN with Storage Stability Additives (CN #2), with a maximum treatment rate of 0.50 vol%. This version of the 2EHN cetane improver is promoted to increase the useful life of cetane improver in a fuel intended to be stored for extended periods.
- 3) DTPB (CN #3), with a maximum treatment rate of 0.50 vol%.

### 2.2.3 JP-8 Additives

In addition to the test additives, all of the FT SPK and HEFA SPK samples were treated with the standard JP8 additives:

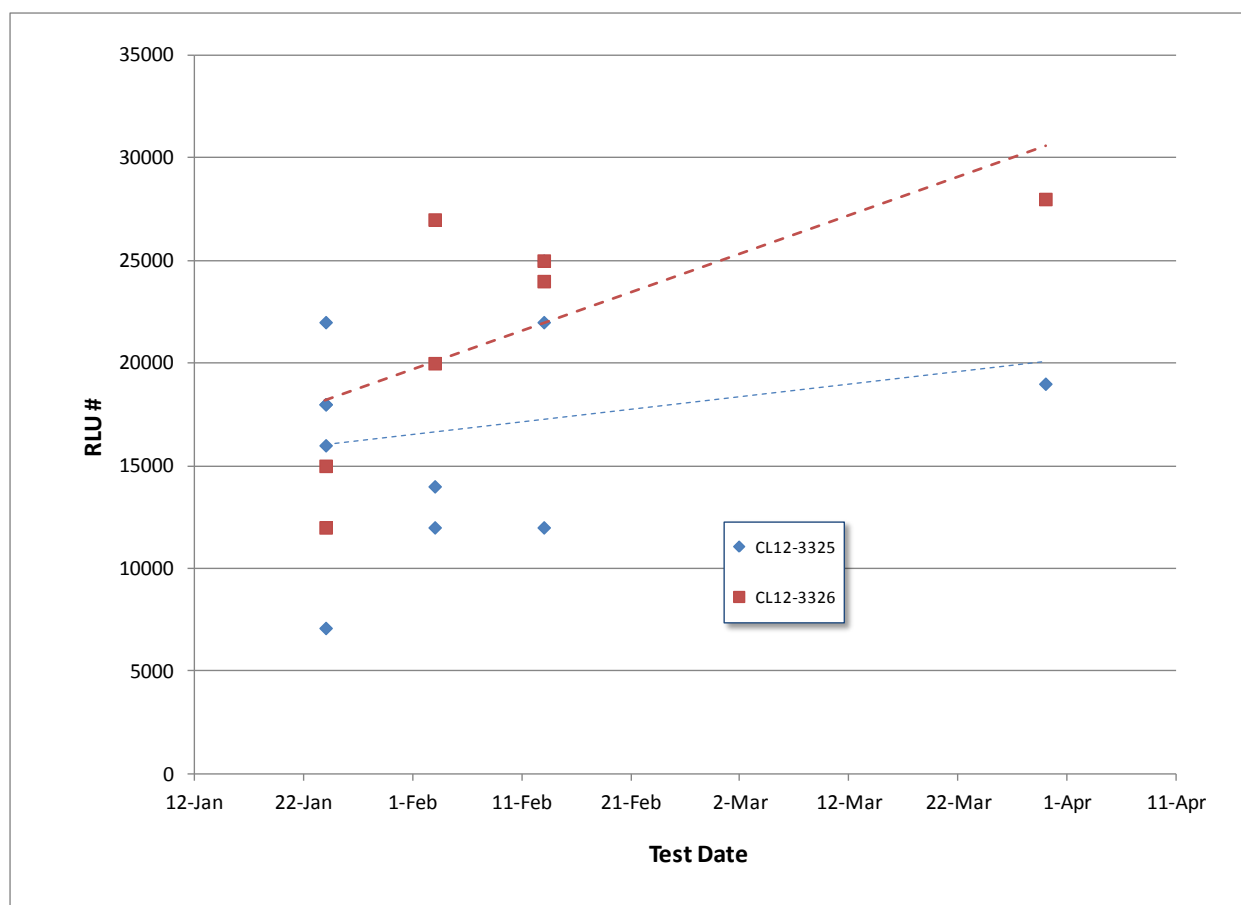
- 1) Corrosion Inhibitor / Lubricity Improver (CI/LI) – this material is dimer/trimers of linoleic acid. It is derived from pine tree resin
- 2) Fuel System Icing Inhibitor (FSII) – Di Ethylene Glycol Methyl Ether (DiEGME).
- 3) Static Dissipater Additive (SDA) – Proprietary chemistry marketed by Innospec as Stadis 450.

The CI/LI and FSII were added to the SPKs in bulk before additizing to the test plan. Since additives can affect the conductivity of a fuel, the SDA was not added until after the blends were sampled for the start of test (SOT) analysis. JP-8 also requires the presence of an antioxidant (AO) but that is also required for the production of SPK so no addition was needed.

It is worth noting that these additives may also have an effect on storage stability. In the ongoing program to convert CONUS to Jet A, work conducted by SwRI [12] and by the Naval Research Laboratory (NRL) [13] suggested that the standard JP8 additive package may be a hindrance to storage stability.

### 2.2.4 Inoculant

The final additive was the inoculant. As soon as the cans were sampled for the SOT (Start of Test) specification tests, they were to be inoculated. To generate a strong live culture to add to the samples, SwRI prepared two culture baths consisting of Bushnell Haas broth to which approximately 100 g of dirt was added. The plan was to grow two batches of potential inoculant, monitor the growth and then use the strongest batch to add to the test samples. The growth of the culture was monitored by the use of the FQS Hy-LITE equipment in accordance with ASTM D7463 [14]. The growth was monitored as seen in Figure 1.



**Figure 1. Inoculant Growth**

When tested immediately after the rest of the additives were in the samples, inoculant batch CL12-3326 was clearly the strongest. Each of the thirty cans received 300 ml of deionized H<sub>2</sub>O and 1 ml of CL12-3326. After this addition, each of the cans was placed in 43 °C storage for incubation.

### 3.0 PROGRAM RESULTS

#### 3.1 BIOLOGICAL PROPERTIES AND EFFECTS

The program plan was to test the samples for biological activity at the start of the test storage (SOT) and then test both the fuel and the water at the midpoint (T18) and then at the end of the 36 week test storage (EOT). The biological activity of these samples was measured with the

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same Hy-LITE system used to monitor the inoculants growth. The numbers in Table 2, Table 3 and Table 4. Biologic Activity in B20 are the same RLU units shown in Figure 1 for the inoculants.

**Table 2. Biologic Activity in FT SPK**

	SOT	T18			EOT	
Sample	Fuel	Fuel	Water	Retest	Fuel	Water
FT-1	21	6	6	32	20	20
FT-2	16	7	9	12	11	14
FT-3	110	7	12	27	13	30
FT-4	13	6	18	18	11	24
FT-5	8	6	22	19	12	13
FT-6	10	8	9	21	17	17
FT-7	56	5	8	26	12	18
FT-8	58	45	9	10	10	11
FT-9	160	7	7	12	20	15
FT-10	15	10	7	17	17	37
FT-11	5	24	7	14	13	8

**Table 3. Biologic Activity in HEFA SPK**

	SOT	T18			EOT	
Sample	Fuel	Fuel	Water	Retest	Fuel	Water
HRJ-1	37	7	19	10	12	20
HRJ-2	7	7	8	11	21	10
HRJ-3	180	6	13	33	14	13
HRJ-4	9	7	10	120	11	20
HRJ-5	23	8	10	22	7	32
HRJ-6	9	7	12	13	9	7
HRJ-7	39	9	16	18	15	36
HRJ-8	15	7	8	16	11	17
HRJ-9	16	6	7	58	26	24
HRJ-10	12	7	7	17	8	17
HRJ-11	17	8	6	16	7	7

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**Table 4. Biologic Activity in B20**

	SOT	T18			EOT	
Sample	Fuel	Fuel	Water	Retest	Fuel	Water
B20-1	36	9	11	36	13	21
B20-2	13	6	12	16	17	22
B20-3	8	5	7	24	21	15
B20-4	12	7	12	14	150	92
B20-5	14	8	45	27	23	31
B20-6	17	5	9	25	21	11
B20-7	8	7	8	97	11	13
B20-8	25	8	11	110	15	83

The results were surprising, until the nature of biological growth was carefully considered. While the low results were not unanticipated for the SOT measurement on the fuel, the low numbers for the midterm (T18) fuel and water tests were a surprise. This caused a fundamental question to be asked: Are the data correct?

It was an important question because the instrument used for the midterm test was not the same as was used for the initial tests. The SwRI unit had failed and FQS, the manufacturer, provided a loaner while it was being repaired. To be sure of the data, before going into discussions of what had occurred and, potentially, why, it was decided to check if the instrument was working properly.

The SwRI lab reviewed the situation with FQS and they walked the SwRI team through the operations step by step. There were a couple of steps which, if incorrectly approached, can result in bad results even though the machine appears to function correctly. This exercise showed that the SwRI technique was potentially flawed. The difference in correct and incorrect is very subtle, thus leaving a question as to whether the data generated was accurate.

The only way to resolve this was to repeat the T18 testing of the water phase. That retesting, under the yellow highlighted 'Retest' in the tables above, showed little effective difference. (The tables are not annotated for additive package because there is no related result pattern.) The EOT testing resulted in similarly low values. The inoculant had an RLU value approaching 30000.

After 36 weeks of 43 °C storage in the presence of supposed excellent food and water, the largest single value is 150 RLU. A visual inspection of several fuel/water interfaces showed that biologic activity started but did not persist. Heat, water and food were present yet no growth. Why?

The working theory is that the materials used in this program simply left the inoculant with insufficient nutrients to grow. A general review of the topic shows that bio organisms need nutrients, particularly those containing nitrogen and phosphorous, to thrive. One particularly interesting report was a study [15] on biofouling of several marine diesel fuels by the U.S. Navy. In this study they were also looking at alternative fuels but they used seawater instead of distilled water. They compared the biological growth in natural seawater, which they state is nutrient poor, and nutrient fortified seawater. The fortified seawater produced more growth. One can posit that if the test was moved to water with no nutrients then biologic growth would be severely curtailed. This is essentially what was done in this test program, so the lack of growth makes sense.

While the program did not generate any significant biomass, it has proved two vital points:

- 1) Fuel, by itself, is not sufficient for biologic growth, regardless of how well it could be metabolized.
- 2) Cleanliness is a vital component in maintaining fuel systems.

By any measure, the fuels in this program should be pretty good fuel for biologic activity. FAME is made from organic fatty acids and, like the fats and oils from which they come, it is biodegradable. The SPKs are primarily linear, with those straight chain carbon to carbon bonds being good for microbial digestion. The fuel by itself, however, is not sufficient for growth.

Typical biologic systems require micronutrients, trace quantities of key elements, for growth. Examples would be organic nitrogen, needed to make amino acids (and thence protein), and phosphorous, for the generation of adenosine triphosphate (ATP) which drives metabolism. Without these key elements growth will not occur.

In this program, micronutrients were provided for the inoculant generation by use of Bushnell Haas broth. What little bioactivity measured in the storage blends was probably a result of the carry over nutrients in the 1 ml of inoculants that was added to each blend, that is from contamination of the water. In real fuel systems, contamination comes from environmental sources. Dirt ingested during filling, dust through vents, the collection of contaminated water, seawater ballasts in ships and barges, poor filtration separation from fuel delivery systems, etc., all combine to provide the nutrients needed to promote biologic growth.

This points to an important concept – keep fuel systems clean. This is not a new idea but one that perhaps needs to be reinforced. If there are no nutrients the contamination will not prosper. An alternative would be to deny the microbes the use of the nutrients. One possible line of study would be to find a way to chemically neutralize the nutrient supply. This might be as simple as a passive device in the fuel system that reacts with one, or more, of the required nutrients. Another would be to find a better way to deal with collected water, particularly for systems that do not allow sump draining. Perhaps a water sequestration additive, such as Aquarius (BASF), could be used to deny the water needed to support biological growth.

### **3.2 CETANE EVALUATION**

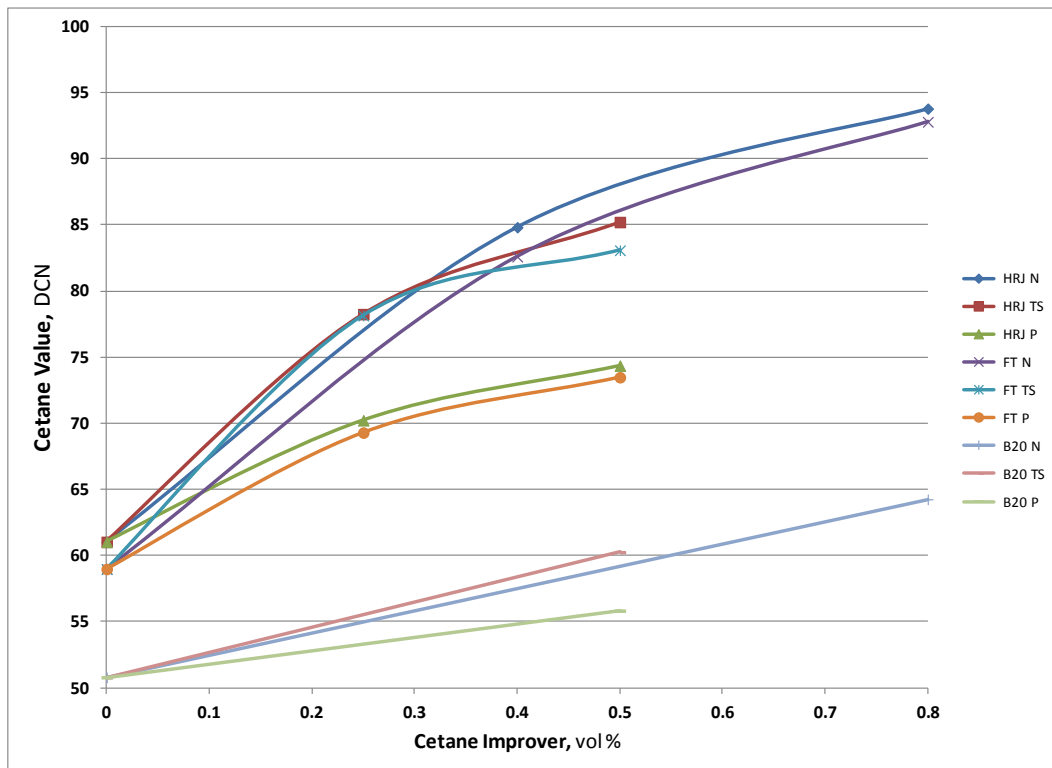
The primary purpose of cetane improver is to get fuel into an acceptable cetane number range. The fuels in this program do not need cetane improver. Both of the SPKs have high cetane values, near 60. Pure biodiesel (B100) is known to have excellent CI properties so it is essentially a cetane improver on its own. As can be seen in the ASTM D6890 [16] test data in Appendix C, these material respond readily to the addition of cetane improver.

The ASTM D6890 test method evaluates combustion quality by measuring the ignition delay upon injecting a distillate fuel into a heated constant volume combustion chamber. The delay is inversely proportional to the Cetane Number and the equivalence is expressed as Derived Cetane Number (DCN). This particular test is also known as the Ignition Quality Tester (IQT) and was originally developed at SwRI.

In preparation for this testing SwRI reviewed the literature regarding the effect of cetane improver on CI fuels. One paper [17] from ExxonMobil researchers was particularly instructive. The paper offers a methodology of predicting the effect of nitrate cetane improver, like the 2EHN used in this program. The paper pointed out two major factors that should be evident in the work from this program if the cetane improvers and the fuels respond normally:

- 1) The higher the base cetane number, or DCN value, the more benefit to be gained from cetane improver
- 2) As more cetane improver is used, the incremental effect is reduced

To evaluate these predictions the data from this program was plotted in the same cetane value as a function of cetane improver addition as appears in the paper. The cetane performance of the test fuels at the start of the program are illustrated in Figure 2.



**Figure 2. Cetane Performance as a Function of Additive Addition at SOT**



In these graphs, Figure 2 above and Figure 3 below, CN #1 (2EHN) is designated “N”, CN #2 (storage stabilized 2EHN) is designated “TS”, and CN #3 (DTBP) is designated “P”. While there are separate plots for all three fuel types, the DCN values for the samples that have identical cetane improver additions but were prepared with and without biocide are averaged.

The test data is consistent with the performance anticipated in the paper. The near 60 SPKs had a DCN rise of over 30 points with CN #1. The B20, starting at just over 50, only increased in DCN value a little less than 15 points with the CN #1. The SPKs show that the effect reduces as the cetane improver amount increases. CN #2, the 2EHN cetane improver with the storage stability additives, followed the same basic pattern as did CN #1. The lesser max treat rate, as dictated by Environmental Protection Agency (EPA) registration limits, resulted in less total efficacy. The peroxide cetane improver, CN #3, followed the same general pattern but at a much lower level of efficacy.

After the storage period, the blends were all retested for ignition quality. Once again this was measured as DCN using the IQT test. The results are plotted in Figure 3.

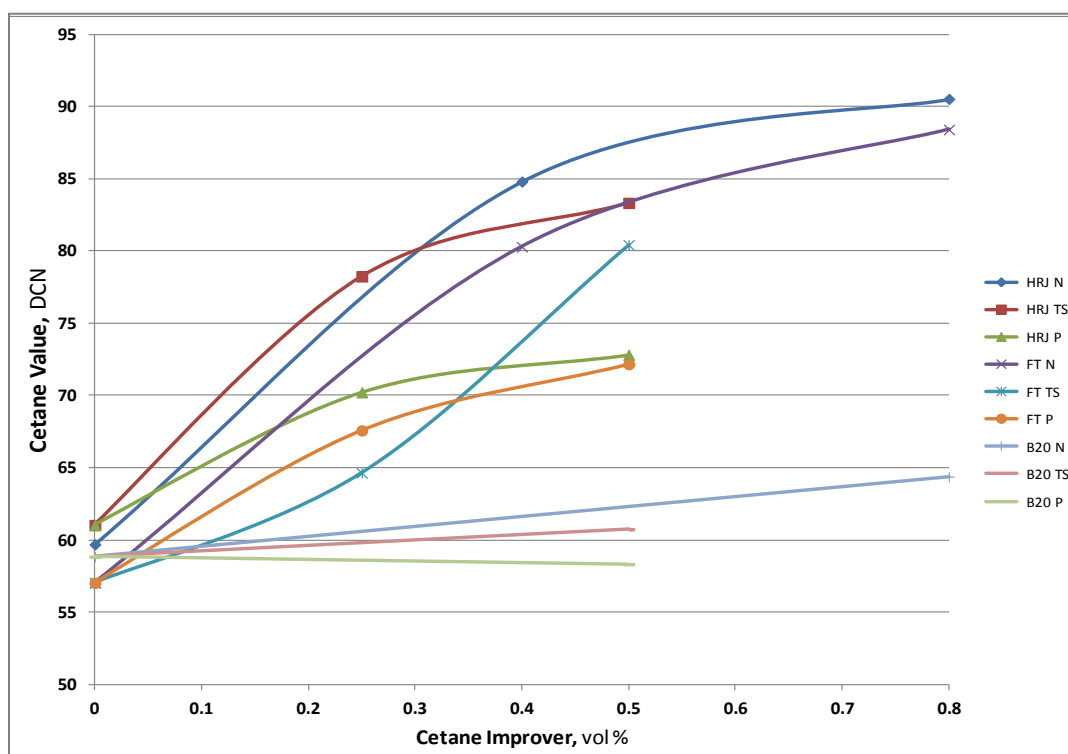


Figure 3. Cetane Performance as a Function of Additive Addition at EOT

The plotted post storage data for the SPKs in Figure 3 above shows a very similar pattern as seen in the pre storage data (Figure 2). This is particularly interesting because the literature for the cetane improvers warn that the material needs to be stored at lower temperatures and used within short order. This program suggests that when mixed into a petroleum product the product is more stable. While there is some degradation of the effective cetane improvement it is still excellent, except for the one sample, FT TS at 0.25 vol%, that has a half dose of CN #2, the 2EHN with storage stability additive. This relative stability may also be due to the nature of the material. The SPKs are just paraffins and, as such, are not as reactive as other hydrocarbons like olefins and aromatics. Before determining if cetane improvers are suitable for long term storage this type of testing would need to be done on a range of normally refined materials.

One point of note was the increase in DCN value, over the storage period, for the two B20 samples that did not include cetane improver. A review of the literature found [18] that an increase in DCN is not unusual for oxidized FAMES. In short duration (less than two hours) oxidation tests, the researchers were able to raise the DCN of FAME by 10 to 15 points. Based on that experience, the increase in DCN for these samples, held at 43 °C for 32 weeks, is reasonable.

As part of the SPK analysis the ASTM D976 Cetane Index [19] was also calculated (see Appendix C). For the FT SPK it reported an average of 56 compared to the neat value of 59. For the HEFA SPK it reported an average of 71 compared to the neat value of 61. That is just how far it missed on the neat material, it has no ability to see the cetane improver. This is not surprising since Cetane Index is simply calculated based on physical properties of the material being tested. In a program on alternative fuels for the U.S. Army [20], SwRI reported that none of the Cetane Index methods tried was able to adequately predict the ignition quality of the alternative fuels being tested.

So the question might be: If cetane improvers are storage stable, could they be added routinely just like CI/LI, SDA and FSII? If the only issue was low cetane value, that might be a possibility. Recent work at TFLRF [21] has shown that excessive cetane value, using the same type of

cetane improvers as in this program, can cause operational problems and significant power reductions. However, there could be a possibility to use cetane improver on an as needed basis. If the ignition quality of a fuel can be accurately gauged, by DCN or actual engine test, then a batch of fuel could be fixed to give better performance. General experience suggests that fuel that has been improved can be used and intermixed with other fuels without an issue.

In general the concern about cetane value is a compression ignition engine issue. Turbine engines do not have the same issue with low ignition quality as CI engines do. However, at a recent meeting of the CRC Aviation Fuel Committee, NASA reviewed [22] work that showed that very low ignition delays, i.e. high cetane values, have a potential adverse effect on fuel nozzle life and combustor stability. Given this, any move to use cetane improver in the field should be preceded by a study of the potential effects on gas turbines. While it is not likely this material would be intended for use with air cavalry units it could be accidentally introduced, but, if approved, the use would be intentional in gas turbines powering the M1 Abrams.

Note: This potential ignition quality issue could lead the aviation industry to introduce controls into the specifications, probably for synthetic turbine fuels. While the U.S. Army has long sought a requirement for ignition quality in the specifications this would not be a direct benefit as the condition described in paragraph above would drive establishing a minimum ignition delay, the equivalent of a maximum cetane value. If the industry were to move to control ignition quality, the best option for the US Army would be to encourage the adoption of an allowable range.

Measuring the actual Cetane Number or DCN would not be readily possible except at the largest facilities as this equipment is not intended for field use. The reason the Cetane Index methods do not work reliably is they only rely on physical properties. The problem is chemistry. SASOL makes an FT SPK in South Africa that is highly isomerized. It has physical properties very similar to the SPKs in this program. Whereas the SPKs in this program have a cetane value of 60, the SASOL FT SPK from South Africa can have a value of 30 or lower. This issue is not limited to South Africa, in the SwRI program on alternative fuels, discussed above, a sample of alcohol derived SPK (ATJ SPK) proved to have a cetane value (by DCN) of 16. To use cetane

improver effectively in the field, there would need to be a chemistry based extension to the Cetane Index concept.

### 3.3 THERMAL STABILITY

For turbine fuel, one of the most critical properties is thermal oxidative stability. Turbine engines run at high temperatures and it is very important that the fuel not cause deposits in the system. The method used for evaluating jet fuel thermal oxidative stability is ASTM D3241 [23], commonly called the JFTOT test. The optimum way of evaluating this property is to do a breakpoint test [24], however that requires three to five determinations per result and was beyond the scope of this program. Therefore the plan was to evaluate the samples in the one test, “Go/NoGo”, specification mode. This required choosing a test temperature.

The specification being used, MIL-DTL-83133G, calls for the fuel to pass 260 °C. These materials, though, are SPKs and the expected production pass requirement is 325 °C. The latter would seem too high for a program including reactive materials like cetane improvers. The former would seem too low given a CRC survey [25] showed that the average Breakpoint for turbine fuel is about 285 °C. Currently, DLA-Energy requires JP-8 to be tested initially at 275 °C and most domestic pipelines [26] require passing at the same temperature for release of Jet A into the commingled batch shipments. Based on these requirements, it was decided to use 275 °C as the JFTOT test temperature in this program. The data for this testing is in Appendix C but to aide this discussion it is compiled in Table 5 below.

Table 5. D3241 Jet Fuel Thermal Oxidative Stability Results

Additives													
Biocide - Kathon FP 1.5	-	100 ppm	-	-	-	-	-	-	-	100 ppm	100 ppm	100 ppm	
CN #1 - 2 ethyl hexyl nitrate (2EHN)	-	-	0.80 vol %	0.40 vol %	-	-	-	-	-	0.80 vol %	-	-	
CN #2 - 2EHN, with storage stability additives	-	-	-	-	0.50 vol %	0.25 vol %	-	-	-	-	0.50 vol %	-	
CN #3 - Di Tert Butyl Peroxide (DTBP)	-	-	-	-	-	-	0.50 vol %	0.25 vol %	-	-	-	0.50 vol %	
FT-SPK Blends			FT-1	FT-2	FT-3	FT-4	FT-5	FT-6	FT-7	FT-8	FT-9	FT-10	FT-11
SOT													
Test Temperature	°C		275	275	275	275	275	275	275	275	275	275	275
ASTM Code			<2	<2	<2	<2	4AP	1	<2	<2	<2	>4AP	<2
Ellipsometry Depth, 2.5mm <sup>2</sup>	nm		6.1	36.4	11.2	25.8	>250	32.4	9.9	15.8	18.1	>250	4.8
Maximum mm Hg	mm Hg		0.1	0	0	0	0	0.5	0.1	0.1	0	0.1	0.1
EOT													
Test Temperature	°C		275	275	275	275	275	275	275	275	275	275	275
ASTM Code			<1	<1	<1	<1	2	<1	<1	<1	<1	<2	<1
Ellipsometry Depth, 2.5mm <sup>2</sup>	nm		21.2	13.2	10.8	10.9	82.6	6.0	46.6	12.9	17.8	95.8	25.9
Maximum mm Hg	mm Hg		0	0	0.1	0	0.1	0	0	0	0	0	0.1
HEFA SPK Blends			HRJ-1	HRJ-2	HRJ-3	HRJ-4	HRJ-5	HRJ-6	HRJ-7	HRJ-8	HRJ-9	HRJ-10	HRJ-11
SOT													
Test Temperature	°C		275	275	275	275	275	275	275	275	275	275	275
ASTM Code			<2	<2	<2	<2	4AP	>4P	<2	<2	<2	>4AP	>4P
Ellipsometry Depth, 2.5mm <sup>2</sup>	nm		8.9	9.7	10.9	12.2	219.4	181.9	4.3	7.3	9.0	>250	>250
Maximum mm Hg	mm Hg		0.1	0.1	0	0	0.1	0	0	0.1	0	0.1	0
EOT													
Test Temperature	°C		275	275	275	275	275	275	275	275	275	275	275
ASTM Code			<1	1	1	<1	1P	2	<1	<1	<1	1P	<1
Ellipsometry Depth, 2.5mm <sup>2</sup>	nm		17.3	26.6	21.2	17.9	130.5	82.4	17.1	15.5	23.6	162.0	8.9
Maximum mm Hg	mm Hg		0.1	0.1	0	0	0.1	0	0	0	0	0.1	0.1
Failing Test		VTR Rating Code 3 or higher, Abnormal (A) and/or Peacock (P). Ellipsometer depth, average over 2.5mm <sup>2</sup> , of 85nm or higher											
Borderline Test		VTR Rating Code 2 or Code <3. Ellipsometer depth, average over 2.5mm <sup>2</sup> , from 75nm to 84.9nm.											

When the fuel was tested initially (SOT), there were several significant JFTOT failures. JFTOT tests are evaluated in two ways:

- 1) The primary evaluation is the visual appearance of the heater tube at the end of the test. The deposit is compared to a color standard with a range 0 to 4. A tube can also be rated Abnormal (A) if the color is a mismatch to the color standard and/or Peacock (P) if there is a multicolored pattern caused by destructive interference of light.
- 2) The test is also monitored for an increase in differential pressure across a test filter that is in line after the heater tube. Any change in differential pressure exceeding 25 mmHg would be considered a failure.

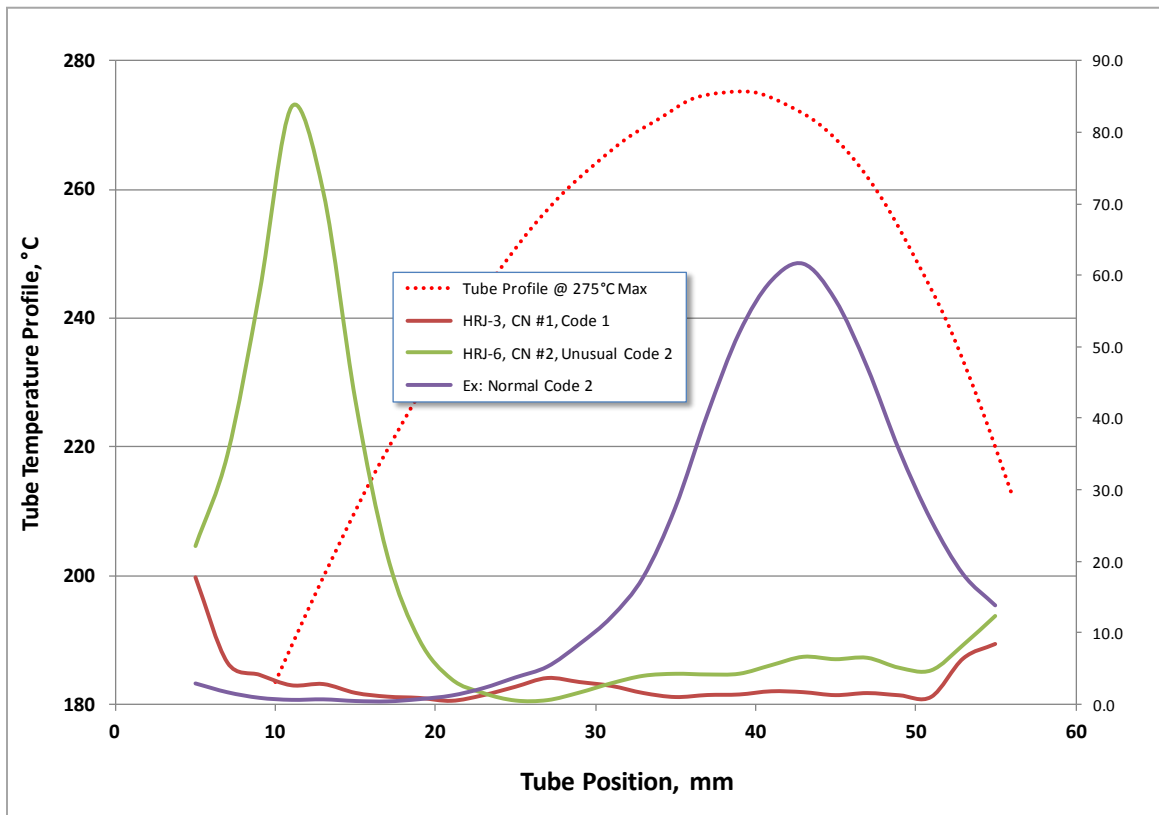
The six failures at SOT were all in the VTR Code 4 category with multiple instances of A and P ratings too. These are about as bad as deposits can get. There were no differential pressure ratings of note. All the samples were retested at EOT and the fuels that failed badly initially now passed the visual analysis. This is not completely unheard of as sometimes very reactive materials form precipitates that come out of the solution with time.

The evaluation of deposits by visual observation has proven successful for quality control over the decades but leaves researchers wanting when trying to evaluate experimental work. For a little over a decade, SwRI has used an advanced technique, ellipsometry, to evaluate JFTOT results. The ellipsometer works by focusing a polarized LASER on the surface of the JFTOT tube. One of the Drude equations (circa 1900) show that the radial azimuth shift of the polarization light will be proportional to the depth of the film through which it passes. It is physically a simple approach but it requires sophisticated equipment. The primary use for ellipsometers is evaluating surface depositions on silicon plates for electronics chip fabrication industry.

Unfortunately, at the outset of this project the SwRI ellipsometer was out of commission so the results were noted but not measured analytically. Shortly after completing the EOT evaluations SwRI received a new ellipsometer and subsequently used it to start evaluating the tubes from this program, starting with the EOT results for the HEFA SPK, in order from HRJ-1.

The first four tubes produced results that matched the visual observations. When the tube for the EOT test of HRJ-5 was placed in the ellipsometer a large deposit signal was observed – at the bottom (cooler part) of the tube. Placing the tube into the VTR, the device used to observe the tube surface, and inspecting carefully revealed an almost invisible deposit. (This inspection resulted in two tube ratings be revised from VTR Code 1 to 1P.) This proved true for five of the six samples that had heavy deposits at SOT. These post test deposits were all unusual being at the bottom of the tube.

The JFTOT uses a resistively heated tube, the test coupon, in a tube in shell heat exchanger. The fuel flows in the annulus and deposit forms on the outside. When a test temperature is specified, 275 °C in this case, that does not describe the whole tube but peak temperature in a tube profile that varies from inlet to outlet. To illustrate why the post test results were seen as unusual the following illustration (Figure 4 below) was generated:



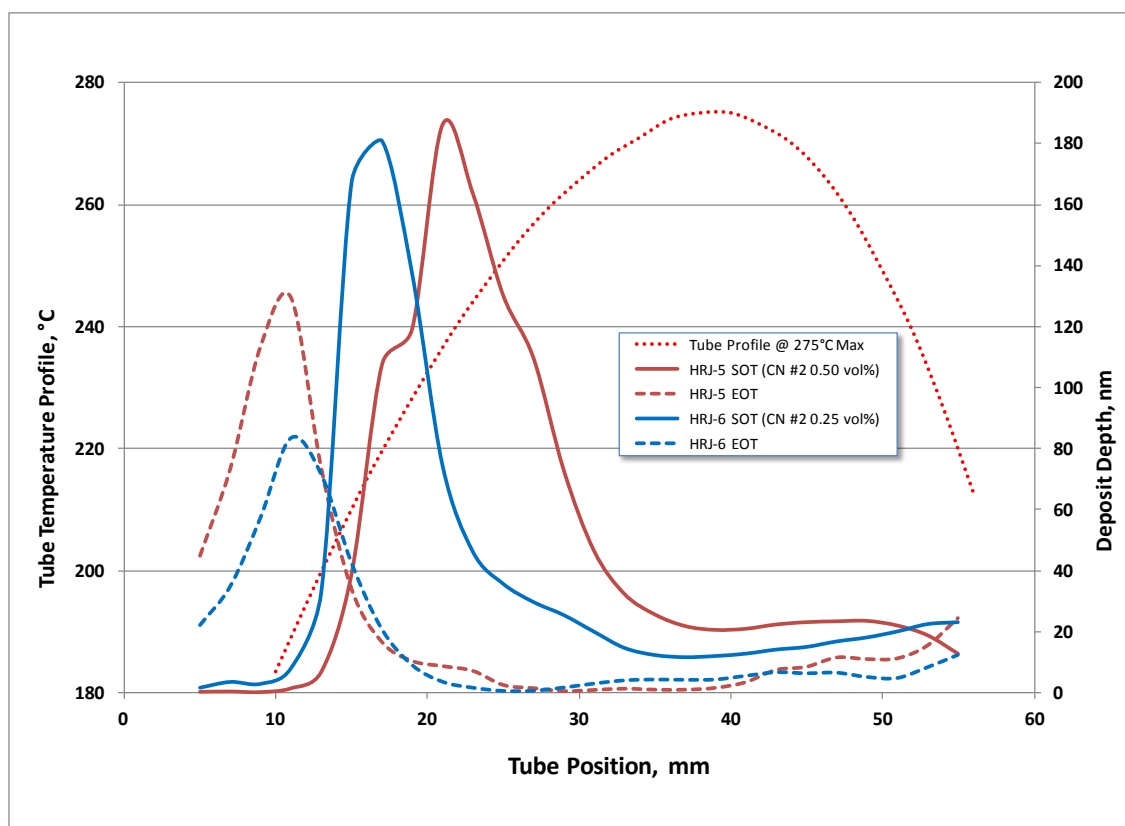
**Figure 4. JFTOT Deposit Placement Comparison**

As part of their development program for the JFTOT, Alcor, Inc. mapped the heater tube profiles for JFTOT tests from 200 °C to 370 °C, the effective range of the instrument. The data for the Tube Profile in Figure 4 above was calculated from data from an archival JFTOT manual [27]. It can be seen that the tube rises in temperature from the inlet and reaches a maximum temperature around 40 mm from the inlet. For comparison purposes, there are three tube profiles (average depth in nanometers, nm, around the tube) illustrated. The first is the profile for HRJ-3, HEFA SPK with the full dose of CN #1, where there is no significant deposit. The second is the profile for HRJ-6, HEFA SPK with a half dose of CN #2, which was rated VTR Code 2. The third is an example of a normal VTR Code 2 deposit.

Typically, JFTOT deposits max out just downstream of the maximum temperature spot, as can be seen in Figure 4 above with the profile of the normal VTR Code 2 deposit. The deposit for HRJ-6, however, peaks at approximately 11mm, approximately 190 °C, on the tube profile. Note that the deposit peaks at about 85 nm. The JFTOT test is rated by judging the deepest 2.5 mm<sup>2</sup> part of the deposit. Visually that was judged VTR Code 2 but the ellipsometer measured that average as 82.4 nm. The current industry consensus is that the ellipsometer depth equivalent to a VTR Code 3 (failure point) is an 85 nm average. This is a really significant deposit and the full dose of CN #2 in HEFA SPK resulted in an average depth of 130.5 nm. Of particular note is that JFTOT deposits are anticipated at the highest temperature part of the tube. Deposits like this at the high temperature spot certainly would be an issue for gas turbines, but having these deposits at such a low temperature part of the tube could be an issue of CI engines too, particularly modern engines designed to run at higher temperature for increased efficiency.

With these data generated for the EOT JFTOT tests, the effort turned to doing the same analysis on the SOT JFTOT tests. (JFTOT deposits are stable for long periods if properly stored.) Following the initial look at the EOT results for the HEFA SPK tests, the first thing investigated was the comparison of SOT and EOT HEFA SPK tests for CN #2. (Figure 5 below).

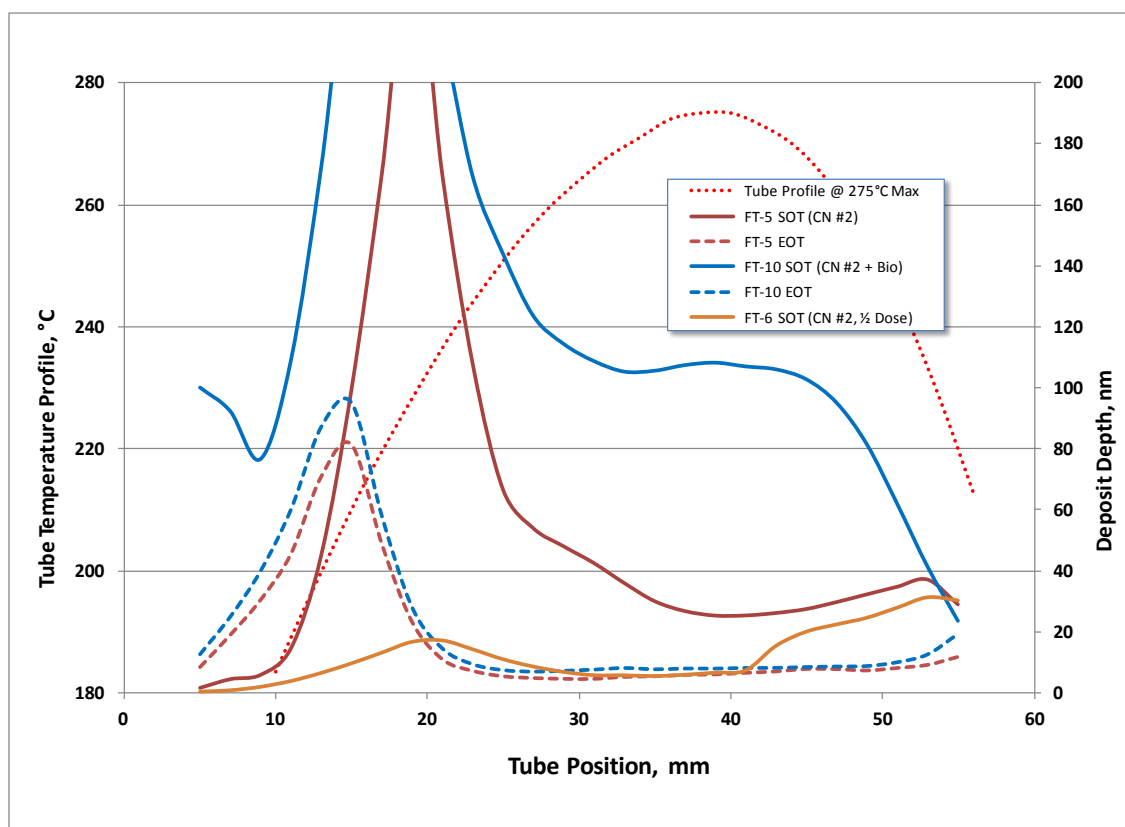




**Figure 5. SOT/EOT JFTOT Deposit Profiles for CN #2 in HEFA SPK**

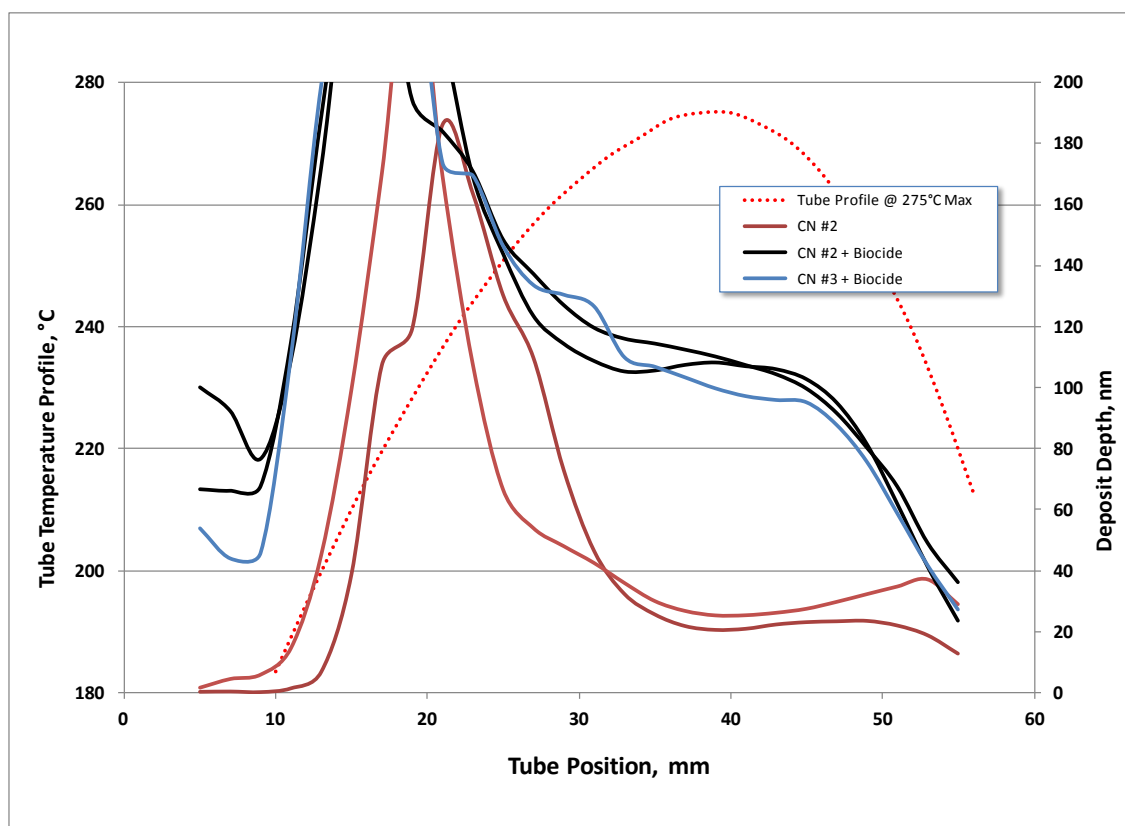
The SOT deposits are much larger than the EOT deposits, not surprising since the former are VTR Code 4 types and the latter are barely visible. The full strength dose of CN #2 produces much more deposit than does the half strength. The SOT deposits are also a little farther downstream than the EOT deposits. The depths of the two SOT tests and the full strength EOT test are where one expects to find Peacock results. Peacock deposits are a result of destructive interference and since the visible spectrum is approximately 380-750 nm the first  $\frac{1}{4}\lambda$  destructive peaks should be in the range of 95-190nm (and repeat as the deposit gets deeper).

As noted in the introduction to the FT SPK (2.1.1), the material was aged and already showed some degradation (high existent gum). Since the FT SPK is so similar to the HEFA SPK it would be interesting to see if it reacted differently than the HEFA SPK. The profiles for FT SPK with CN #2 are in Figure 6.



**Figure 6. SOT/EOT JFTOT Deposit Profiles for CN #2 in FT SPK**

A different fuel produced different, bad results. The full strength SOT CN #2 deposit is much heavier for FT SPK than for HEFA SPK while the EOT result was smaller. On the other hand, the half dose test was much reduced (even though it is a significant amount). This kind of difference is not unheard of in JFTOT testing and is one of the reasons why the preferred research technique is Breakpoint combined with ellipsometry. It could be that the test is on the edge of failure, with some (most in this case) blends failing badly and the occasional passing. One thing noted in this analysis was the large increase in deposit when the Biocide was present. This prompted a comparison of the heavy deposits with and without Biocide in Figure 7.



**Figure 7. Comparison of Heavy SOT Deposits W/O Biocide**

In Figure 7 above the intent is to compare general results so the SPK deposits are identified and color coded by the additive package. Clearly the addition of Biocide in the presence of CN #2 has a synergistic effect. The resulting deposits are substantially heavier than with CN #2 alone. (This is one of the important benefits of ellipsometry, the ability to quantifiably differentiate deposits which, by VTR Color Code, are otherwise indistinguishable.) At initial review, the thought might be that since CN #2 has shown to be problematic for this, and other reasons, this is an interesting but moot point. True except for the fact that the same synergistic pattern appears for one test with CN #3, the DTBP cetane improver, that otherwise had no significant JFTOT deposition.

Clearly, from this testing, CN #2 is not suitable to be considered for an additive for U.S. Army field use. If an additive is approved for field use it might find its way into an aviation application.

While there might not be the intent to qualify a cetane improver for use in aviation applications there should at least be reasonable assurance that it will not cause harm if introduced accidentally. Even if it could be guaranteed that CN #2 would not get into aviation applications the relatively low temperature at which this material deposits could be a problem for ground based applications, particularly modern diesel engines and the gas turbines in use. On the other hand CN #1 might be a candidate for qualification for field use. Passing thermal stability screening is a key step for approving any aviation additive. Like in the discussion of cetane stability (3.2), this would need to be evaluated, by Breakpoint, in a broader range of refined turbine fuels.

While, except for one sample with the Biocide, CN #3 produced little in the way of deposits, the idea of a peroxide additive would be difficult to justify for aviation use. Peroxides are seen as a substantial hazard to elastomers in aviation applications. Perhaps the tertiary structure of the alkyl constituent groups alleviates that problems but it would have to be proved beyond a doubt. Considering that 2EHN, as used in CN #1, is a superior cetane additive, a DTBP additive should only be pursued if 2EHN proved problematic in testing.

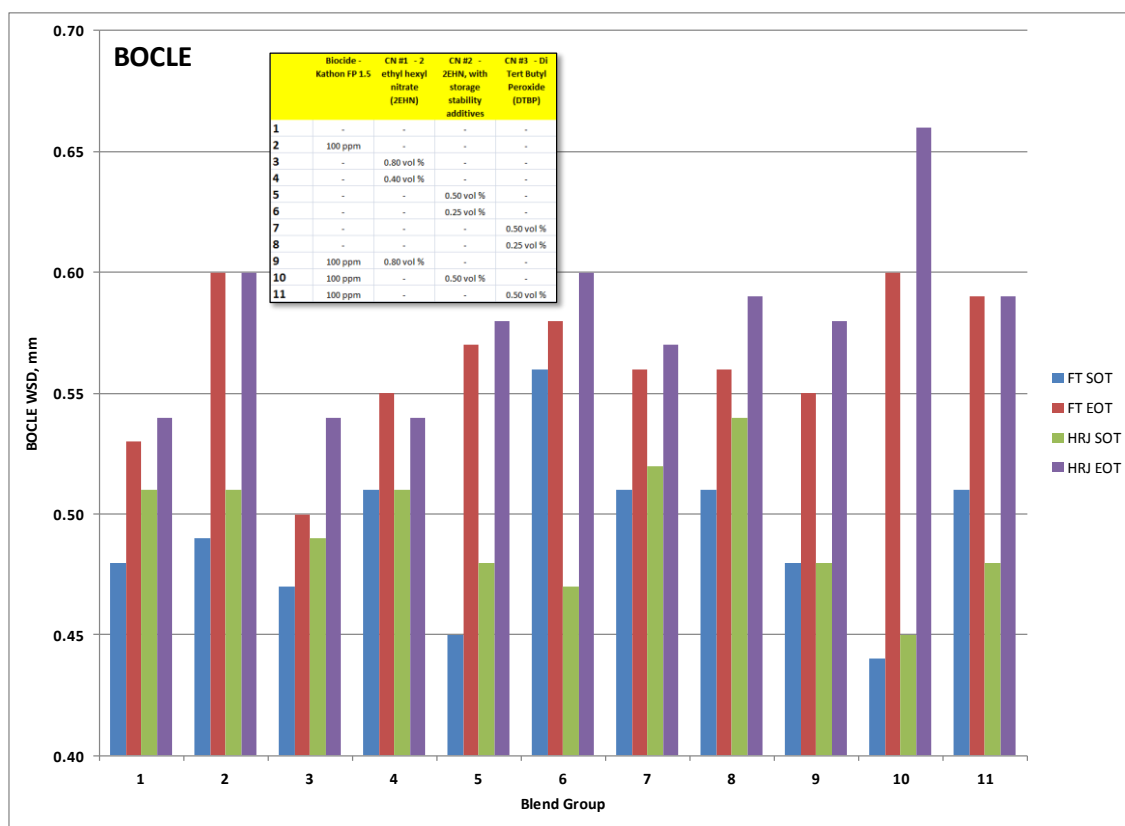
Approving an additive for use, even on an intermittent basis, in an aviation turbine fuel would be guided by the principles of ASTM D4054, 09 version or later [28]. The aviation industry developed that standard as a means to direct the proper steps in developing a new aviation fuel or additive. Like all standards it is always under review. This program has revealed a serious shortcoming in the methodology. Currently, an additive has to be evaluated for thermal stability effects and for additive to additive compatibility, but separately. This testing has shown the potential for synergistic adverse effects on thermal stability. This is a critical item that needs to be discussed in the industry. After decades of having a limited selection of turbine fuel additives there is a push to adopt new additives materials and types. While this work is primarily commercially driven it is vitally important to the US military in light of the pending DLA-Energy switch to purchasing Jet A, in place of JP-8, in CONUS.

### 3.4 LUBRICITY

The lubricity data in Appendix C shows another area where there is a notable difference before and after the storage period. The SPK blends were tested by the BOCLE (Ball On Cylinder Lubricity Evaluator) test, ASTM D5001 [29]. The B20 blends were tested by the HFRR (High Frequency Reciprocating Rig) test, ASTM D6079 [30].

The BOCLE test works by running a stainless steel ball linearly, under load on a hardened cylinder. This has been shown to reasonably represent the sliding type of motion typical in an aircraft fuel pump. There is no official specification for lubricity for commercial Jet A/A1 in ASTM D1655, only a note that turbine fuel is expected to have a wear scar diameter (WSD) of less than 0.85mm. JP-8 does not have a requirement either but the specification requires the addition of CI/LI. The qualification process for the approved CI/LI requires it be able to reduce the WSD of clay treated Isopar M (a commercial isoparaffinic solvent) from greater than 0.85mm to less than 0.65mm. (Some believe this means that the military specification is a WSD of less than 0.65mm for JP-8 but it does not.) The only specifications that require the WSD to be below 0.85mm are UK(MOD) DS91-91, for highly hydroprocessed turbine fuels, and ASTM D7566, for blends of semisynthetic turbine fuel.

The SPKs in this program would have very high WSDs if not treated with CI/LI but this program included addition of the normal JP-8 additive package to these blends. As a result, all of the blends start off with excellent lubricity as seen in Figure 8.

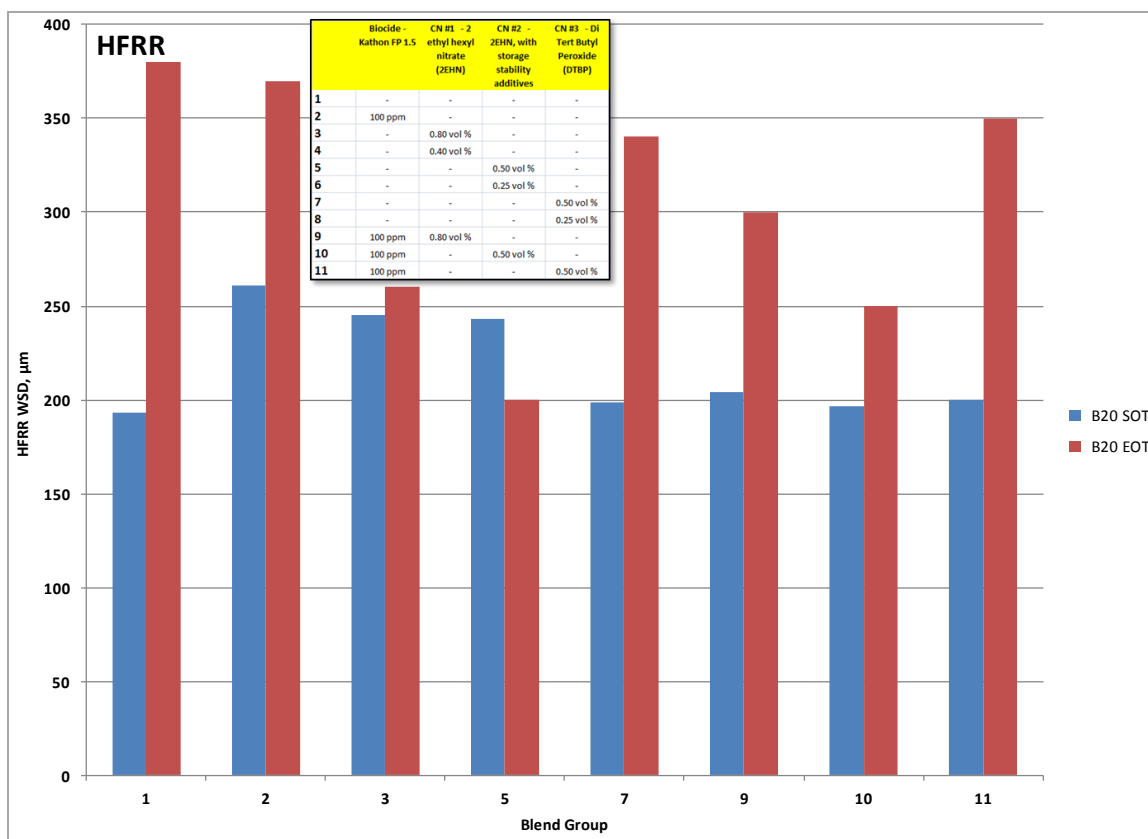


**Figure 8. SOT/EOT BOCLE Results for FT SPK and HEFA SPK Blends**

The data in Figure 8 above shows scatter in both the SOT and EOT results. (The Blend Group additization schedule is in the inset.) Is it significant or just an artifact of the methodology? In Appendix D there is a statistical review of the various methods. The scatter in these data is far in excess of the reproducibility expected for this method. The neat fuels, Blend Group 1, show the effect of aging. CN #1 and CN #3 show similar response (Blend Groups 3/4 and 7/8 respectively). CN #2 (Blend Groups 5/6) shows a slight advantage initially but in the end performs worse than the neat material and the other two additives. Once again this limited amount of testing with SPK does not constitute proof that 2EHN or DTPB would have no harm in storage but neither does it indicate any particular problem. More comprehensive testing with a variety of refined fuels would be required to confirm suitability.

Special note should be taken of the four Blend Groups (2, 9, 10 and 11) with Biocide. While they all perform similarly to the groups with equivalent cetane additive at SOT, the WSD results at EOT are uniformly worse with the Biocide present. While none of the results were out of the de facto limit of 0.85 mm WSD, one sample did exceed 0.65 mm WSD. That was with additized fuel that started with very good lubricity. Not all fuels respond as well to CI/LI as evidenced in this test so there is a possibility that the same extended storage effort, with Biocide, on a different fuel could result in a severely hardened fuel.

The HFRR test works by reciprocating a stainless ball, under load across a hardened plate at a high frequency. This has been associated with the cyclic motion typical of diesel engine injectors. The B20 specification limits the HFRR WSD to 520  $\mu\text{m}$ , the same as for regular ASTM D975 (31) diesel fuel (neat to B5). With the prevalence of ultra low sulfur diesel (ULSD), regular diesel usually requires the addition of lubricity additives. FAME, however, is chemically very similar to the kind of materials used as lubricity improvers. Thus, it would be anticipated these blends would have excellent lubricity and, as seen in Figure 9, they do.



**Figure 9. SOT/EOT HFRR Results for B20 Blends**

*Note: the group numbers in Figure 9 above are the same as in Figure 8. Because there were no half dose blends of the cetane improvers for the B20 samples there is no data for groups 4, 6 and 8. This makes it easier to compare group to group in these two graphs.*

The differences between SOT and EOT in the B20 tests are not as dramatic as with the SPK. Certainly there is change, as might be expected by the same oxidizing process that results in higher cetane values and the variation chemistry is discussed in the same paper [18]. There is a mild correlation between EOT acid number and the change in HFRR rating, ( $r = 0.5750$ , less than 95% confidence at seven degrees of freedom).

The 2EHN additives seemed to suppress the loss of lubricity unless Biocide was present. Biocide did not have the stand out negative impact as with the BOCLE rating in the SPK samples but it still appeared to have a negative effect. The combined HFRR and BOCLE results for the samples



with Biocide reinforce the understanding the Biocide should be only be used on an intermittent basis to cure specific problems and not be used as a general preventative.

### 3.5 OTHER RESULTS OF NOTE

In this section other data points needing discussion will be covered. These discussions may be from direct observation of the data tables or from the analysis of method statistics in Appendix A.

#### 3.5.1 Conductivity

As noted in the section on JP-8 additives (2.2.3) the conductivity additive was not put in until after the blends were sampled for SOT testing. This was because some additives can impact the conductivity too. This proved to be true as CN #2, the 2EHN with storage stability additives, had a significant impact on the initial conductivity of the samples. None of the other additives had a significant impact so only the CN #2 results are plotted in Figure 10 below.

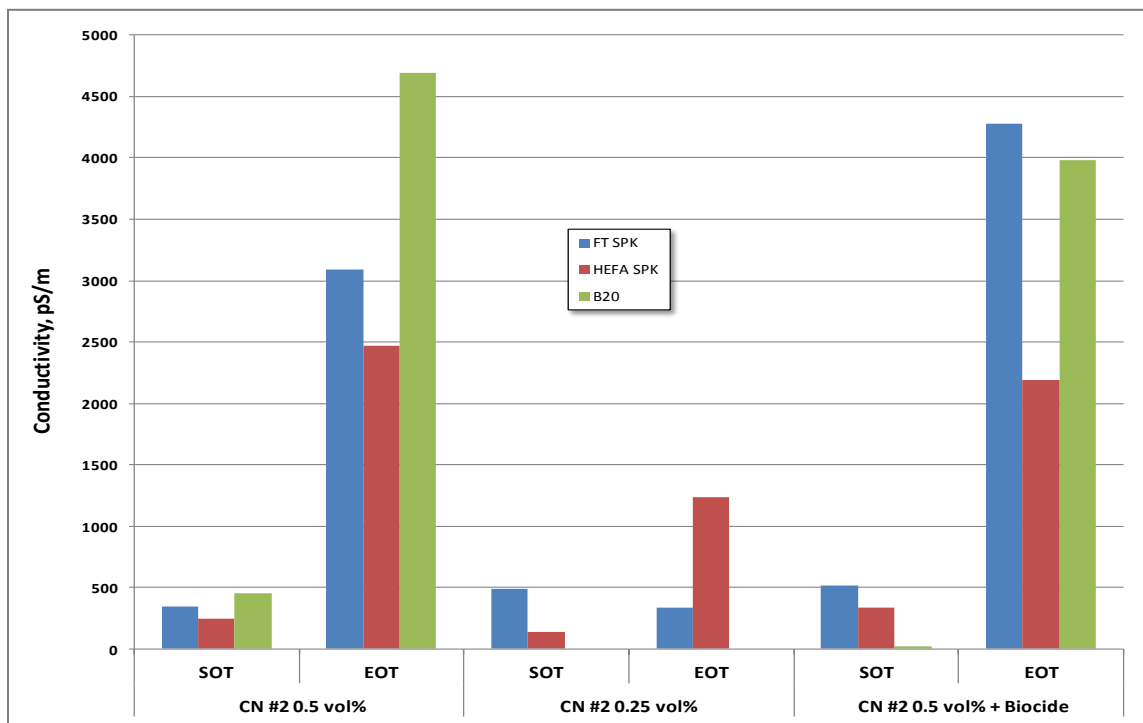


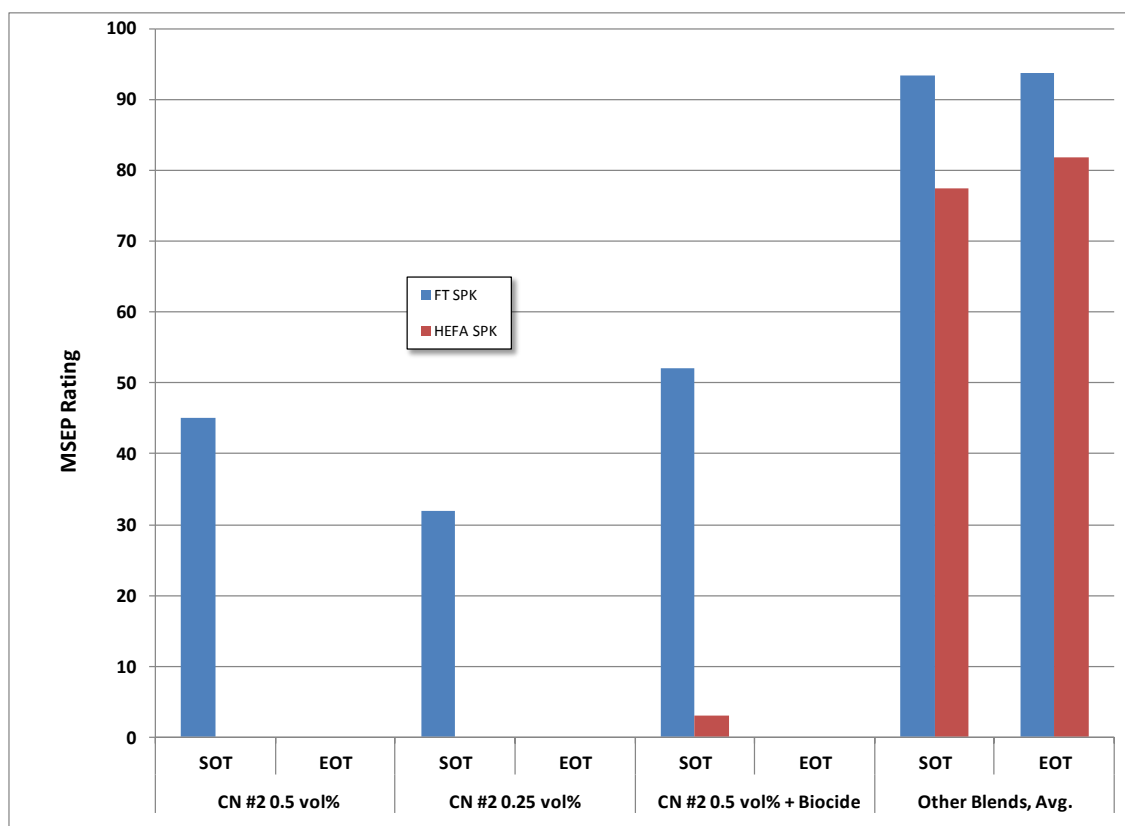
Figure 10. Effect of CN #2 on Conductivity

The SOT testing showed that CN #2 does impart conductivity to the fuels, except for one instance with the B20. The values did not exceed the typical limit, 600 pS/m, for fuel with conductivity improver. The real surprise came at the end of the storage. Conductivity improver has a limited life so it was not surprising that the blends not containing CN #2 were low; 38 (pS/m) average for the FT SPKs, 78 average for HEFA SPK and 145 average for the B20s. The results for the blends with CN #2 (see Figure 10 above) were astonishing.

In all but one case, the EOT conductivity data for blends containing CN #2 was significantly higher than the SOT conductivity. As can be seen in Figure 10, among the seven, of eight, samples with the high response the lowest response was to more than double the standard limit (1239 pS/m versus 600 pS/m). The ASTM electrostatic guide (32) states: “The maximum limits prevent adverse effects on aircraft fuel capacitance gages.” Capacitance gages are used in automotive applications too, so this could be an issue ground vehicles directly and a potential problem for aircraft if the fuel was accidentally dosed with this additive.

### **3.5.2 Water Separation**

The water separation characteristics of the SPK blends was evaluated as MSEP rating in accordance with ASTM D3948 [33]. This device has a small filter made from materials similar to those found in aviation filter separators. The rating is a form of turbidity measurement with 100 being totally transparent and 0 nearly opaque. Except for the one sample of HEFA SPK with CN #3 + Biocide that got a MSEP of 58 (and also failed the JFTOT test) the problem results were with the samples containing CN #2. As with the conductivity analysis (Figure 10), the data displayed on water separation in Figure 11 below is focused on the samples containing CN #2.



**Figure 11. Effect of CN #2 on Water Separation**

The effect of CN #2 is dramatic. The FT SPK was slightly more resistant to the additive at SOT than the HEFA SPK but by EOT they were equally bad. The samples with CN #2 became cloudy over the course of the storage period. This material was evidently a stable suspension because, since the cans contained water bottoms, the blends were carefully decanted to avoid dispersing water into the sample. The sample did clear up when passed through the JFTOT pre-filter, which suggests that the suspension contained the materials responsible for the heaviest deposits (at SOT) in that testing (3.3).

Unknown in this program is the effect of these additives on the water separation characteristics of the B20 blends. The standards for Biodiesel do not require an equivalent test but the ability to separate water is important in CI applications too. One of the known issues with biodiesel is water separation. Improved production quality control has reduced that issue but it is still a concern. The limitation to specifications is that they are minimum amount of tests that need to be

done to assure the product produced is correct, they do not test every performance criteria of a material. The problems seen with the water separation characteristics with SPK suggest any plan to approve an additive needs to include an evaluation of water separation in CI fuels too, both as refined and biodiesel blends.

### 3.5.3 Rancimat Testing

The Rancimat test [34] is a forced oxidation test that evaluates the oxidative stability of biodiesel blends. (The name comes from the device used, an instrument originally designed to evaluate cooking oils for rancidity.) It relies on bubbling air through a heated sample and then monitoring the effluent gas for ester decomposition products. The time it takes to reach the limiting value is called the Rancimat Time and the minimum expected period is six (6) hours.

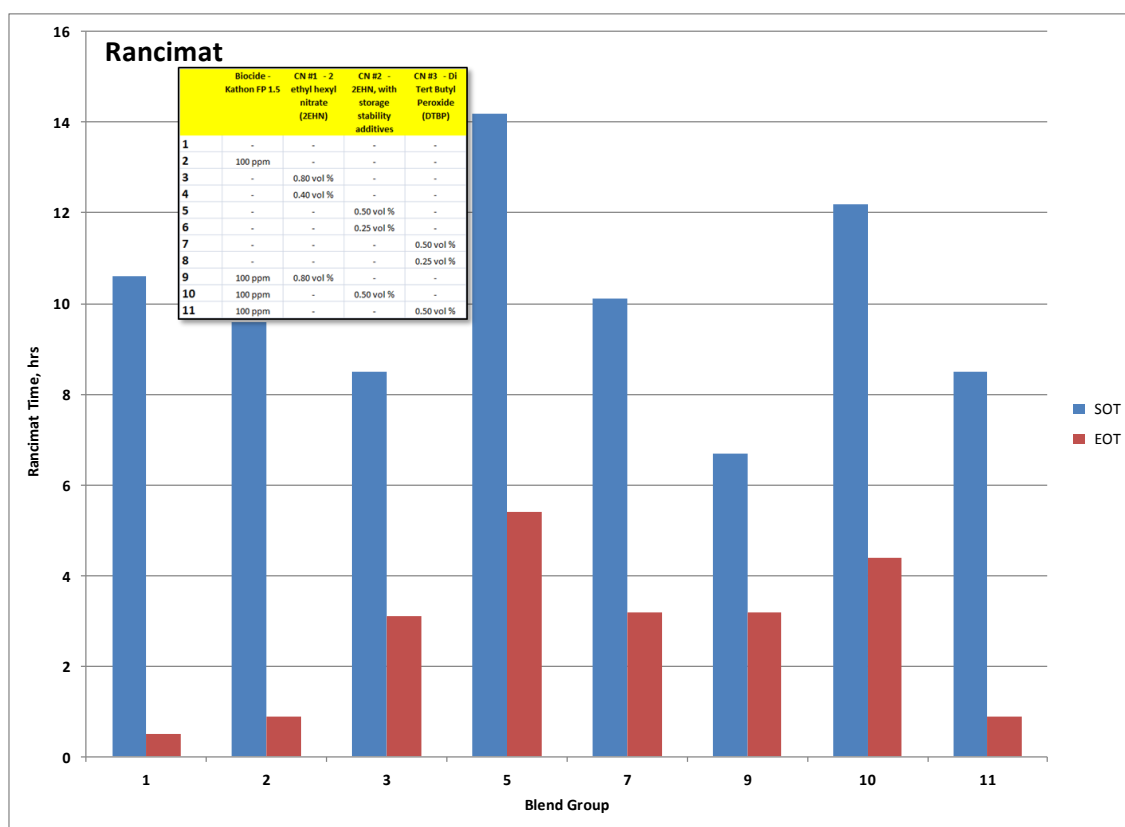


Figure 12. SOT/EOT Rancimat Results for B20 Blends

The EOT results in Figure 12 above show that storing biodiesel for long periods can cause problems. In general, the stability was better with the cetane improvers than without. The storage induced an increase in acid number for all of the samples. There is a statistically significant (95%) correlation ( $R = 0.7127$ ) between the increase in acid number and the loss in Rancimat value. By comparison, the acid number values for the SPKs went down during the course of the storage period.

This is the only test that the blends with CN #2, the 2EHN with storage stability additive, performed better than the other samples. It increased the Rancimat at SOT and was the closest to passing at EOT. This confirms that the storage stability additive does improve storage stability but the adverse results in so many other tests should discount any thought of using this additive in the field. Also of note is another instance where adding the biocide generated poorer results.

### **3.5.4 Statistical Analysis of Data Precision**

In Appendix D the results from every test are compared to the expected precision for the method. The details of the analysis are discussed there but following are some points worth noting. The test methods that showed the biggest effect from the presence or lack of additives were obvious by inspection and have been discussed. There were other methods that produced acceptable results but the data exceeded the acceptable precision, as described in the Appendix D.

In Table D-1. Data Analysis Precision Review for Jet Fuel Type Samples, the following should be considered:

- 1) The estimated precision(s) for the automatic freeze point test exceeds the what might be expected for the manual method, if the fuels were identical. Freeze point is a test known to be contamination sensitive. There is a hint in the data that CN #2 may be an issue but not to the extent that its problems have been seen in the testing previously discussed.
- 2) Fuel color is a 'Report' item for JP-8 without a limit. The precision data suggests there is an issue and an inspection of the data shows that CN #2 routinely causes fuel darkening. That is an important aspect for handling purposes. While color is not a limited

specification item, its change from point A to point B can be seen as evidence that a change has occurred and trigger a time consuming analysis of fuel. Knowing that an additive colors a fuel would provide an explanation that could preclude expensive testing and excessive time delays.

- 3) As noted in the introduction to the SPK fuels (2.1.1), the FT SPK showed its age by its high existent gum level (that even exceeds the MIL-STD-3004 intra-governmental transfer limit). The precision analysis shows that it is far from the expectation for this test if all of the fuel samples are identical. Once again, the biggest effect is from CN #2 which consistently produces failing values. It is not unheard of, however, for performance additives to cause an existent gum problem. The prime example would be the JP-8+100 additives used by the U.S. Air Force on some high performance aircraft. The appropriate evaluation then would be to determine if the residue was solvent soluble.

In Table D-2. Data Analysis Example for B20 Type Samples, the following should be considered:

- 1) The analysis of the aromatics testing, by ASTM D1319, was a problem. There is no data for the SOT level because the SwRI laboratory that runs the method declined to run biodiesel, saying it was not appropriate. A discussion changed the policy but not before the test material had been disposed. The EOT analysis worked and the data was reasonably in control. A look at the data, though, brings up an interesting point. The method is only used to test aromatic content but it also reports olefins and saturates. Together they add up to 100% but since this is a B20, that is not possible. The specification, ASTM D7467, does not discuss this fact and only mentions that the precision has not been established for biodiesel blends. The working hypothesis is that the FAME is held back in the test column and the measurement reflects only the hydrocarbons.
- 2) The estimated precision for the Ramsbottom Carbon method, ASTM D524, is far in excess of what is expected. The scope of the method says that nitrate cetane improvers, like CN #1 and #2, can result in high readings but the data does not show a definitive

corresponding pattern. The method is not scoped for FAME so that may have an unknown impact on the results.

- 3) The FAME content measurement, EN 14078, is in control, with data better than expected for repeatability, but there is a near 6% increase in reported FAME concentration. With the testing this well in control, the data shows a real impact of storage. Since the EOT evaluation is just as well in control as the SOT evaluation, the issue has to be related to the FAME content and the effect of high temperature storage thereon. The assumption is that this is part of the same chemical process that generates higher cetane values in the blends made without cetane improver.

Appendix D notes that the comparison of the estimated precision, based on data from this program, was bounded by two times the method precision at the reproducibility level. Testing results from the ASTM Inter Laboratory Crosscheck Program (ILCP) program often show methods at this level for reproducibility for tests on the same sample. Since these are, by definition, different samples, using the higher reproducibility bound allows identifying the tests where the additives have a important effect.

## 4.0 CONCLUSIONS

In progressing through the report the following points stand out:

- The biocide and cetane improver additives are potentially dangerous and require careful handling. In the concentrated form the biocide is a toxic agent and would require specialized personal protective equipment for handling. The cetane improvers are subject to explosive decomposition if exposed to excessive heat. These issues probably would preclude routine use except by appropriately trained personnel.
- Because the misuse of biocide could lead to an incurable biological infestation it should be limited to 'as needed' use.

- If a fuel system is kept clean and free of nutrients, biologic growth probably will be kept in check regardless of how ideal the fuel is as a food supply.
- The neat cetane improvers in this program proved to be reasonably stable, based on this limited experience. The problem for field use is there is no ready method to evaluate the cetane value and over boosting it, exceeding a value of 60, has been shown to cause operational problems.
- In thermal oxidative stability testing, the plain 2EHN (CN #1) cetane improver passed the testing without any evidence of a deposition problem. The version with storage stability additive (CN #2) failed at temperatures that would likely be a problem for both compression ignition and turbine engines. The peroxide additive (CN #3) had mixed results so, combined with the fact it is less effective as a cetane improver, it would not be the first choice for additional testing.
- The visual rating methodology used in the JFTOT test is meant for evaluating fuel deposits, relying on the typical chemistry of turbine fuels. Deposits generated from other chemistries may not be readily apparent so using more sophisticated approaches to deposit measurement, such as the ellipsometer at SwRI, is critical to assuring there will be no deposition problems.
- The evidence of additive related synergistic antagonisms in fuel deposition is a revelation. The current industry practice for evaluating additive to additive compatibility is limited to a hot and cold solubility/miscibility evaluation.
- The use of 2EHN cetane improver did not prove to have a negative effect on the lubricity, on blending or after extended storage, for either the SPK or the B20.
- The very adverse impact of the Biocide on the lubricity of the SPKs, after storage, is a clear warning that this material should not be used for long term storage. The negative effect on the B20 was not as pronounced but it was there.
- The exceptional, storage driven increase in conductivity of the test blends containing CN #2 is additional proof it may not be suitable for use in the field, where storage is a



potential issue. Another lesson learned is the importance of evaluating the storage effects on additives.

- The immediate bad effect of CN #2 on water separation characteristic would be a major impediment to approving its use in turbine fuel, as it is in the base JP-8 specification. Even if it were not, the aviation industry has developed a fit-for-purpose guide to approving fuel components and additives that examines the complete technical use and handling of turbine fuel. The lack of a fit-for-purpose guide for CI fuel makes the reasonable examination of a proposed alternative fuel component or additive guesswork.
- Conducting statistical analysis of test precision is a good way to deduce additive effects that may not be sufficient to render the material off specification. It is also a good technique for considering the appropriate application of methodology.

These points can be consolidated to the following overall conclusions:

- 1) Biocide should not be used on a continuous basis or as a storage preservative. The potential to generate an incurable infestation and the demonstrated adverse effects of fuel properties resulting from long term storage are deciding factors. The preferred alternative is to keep systems clean and dry. Microorganisms need three things to prosper; food, nutrients and water. Regardless of how good the food supply, the system will remain clean if one or both of the other requirements is denied.
- 2) Cetane improvers should not be used as a continuous use additive like CI/LI or FSII. Continuous use additives require some form of standard addition whereas cetane improvers are usually titrated to achieve the desired effect. Note, however, that since DLA-Energy buys commercial diesel fuel for the U.S. Army, some amount of cetane improver is already being consumed.
- 3) Given that the U.S. Army has cumulative, if inadvertent, experience with cetane improver, it is reasonable to consider intentional application for specific purposes. An example would be to assure adequate cetane value for JP-8 used with the U.S. Army's MQ-1C Gray Eagle, the CI powered version of the Predator. Based on the that potential

use of the data from this program can be used to give a preliminary evaluation of the additives:

- a. CN #1, the 2EHN cetane improver – In the course of this testing this additive has performed well. Based on the limited fuel selection, there are no standout problems that would preclude its use as an additive.
  - b. CN #2, the 2EHN cetane improver with the storage stability additive – In the course of this testing this additive has performed poorly. It should be noted, however, that this additive (in particular the storage stability component) was likely formulated for normal diesel fuel with the typical aromatic content. It did not perform as poorly with the B20 as with the SPKs.
  - c. CN #3, the DTBP cetane improver – In the course of this testing performed moderately well. The biggest problem is the lack of efficacy compared to the nitrate additive. Its use would require a large logistics trail and handling effort.
- 4) The biggest impediment to successful use of cetane improver on an as needed basis is the lack of a simple test to evaluate cetane value. The only accurate methods, the actual cetane engine or the continuous volume combustion chamber instruments, are expensive/complex installations that will only be found in the largest labs. They would not be appropriate for U.S. Army's PQAS system, for instance. This lack would make it hard to tailor a narrow application of cetane improver for a system like Gray Warrior.
- 5) The broad nature of this program has generated a critical observation regarding additive to additive compatibility that is not currently being considered in the ongoing approval programs. Currently the compatibility is judged primarily on miscibility/solubility at low and high temperatures. The synergism between the biocide and the cetane improvers that produced massive increases in JFTOT deposition would not have been found by standard practice.

## 5.0 RECOMMENDATIONS

Based the knowledge gained in this program the following recommendations are offered :

- 1) On the basis of the potential hazards and negative outcomes, the biocide additive should not be approved for routine use. Biocide is important for cleaning systems as needed but that should handled by specially trained personnel.
- 2) As an alternative to excessive use of biocide, consider a program to develop a means, active or passive, to deny the nutrients and/or water to potential microbial growth.
- 3) If there is any intent in using cetane improvers in the field then storage stability testing needs to be conducted with normally refined petroleum fuels too. This to prove the stability is due to the dilution into fuel in general and not just because it was diluted into relatively unreactive SPK.
- 4) Approving cetane improver for field use will require the study of the effect on gas turbines for intentional use with tanks and accidental use in helicopters. The best cetane improver to study/promote appears to be the 2EHN type. The most flexible plan would be to get it approved as a general use additive that could be in all equipment, surface vehicles and aircraft. The next step would be to evaluate the basic properties in a typical refined jet fuel, to be sure the observations in the program hold when aromatics are present, and then proceed through the fit for purpose program as describe in ASTM D4054.
- 5) For maximum flexibility, there needs to be a way to assess cetane value in the field. The existing Cetane Index methods cannot see the chemistry issues that drive the differences in ignition quality so a means of evaluating that chemistry needs to be developed.
- 6) With a successful demonstration in the thermal stability testing with SPKs, the next step would be to demonstrate that a 2EHN based cetane improver would cause no thermal stability issues in a broader range of refined jet fuels. If that effort is successful, then a larger effort could be dedicated to evaluating the general fit for purpose properties with the idea developing a qualified additive that could be used by the U.S. Army in both ground and air applications.

- 7) Since the U.S. military will become increasingly dependent on commercial turbine fuels with the switch to Jet A from JP-8, the issues with nearly invisible heavy deposits in the JFTOT test and the synergistic effects on thermal stability with mixed additives needs to be discussed within the aviation industry.
- 8) With a successful demonstration in the lubricity testing with SPKs and B20s, the next step would be to demonstrate that a 2EHN based cetane improver would cause no thermal stability issues in a broader range of refined jet fuels and diesel fuels. If that effort is successful, then a larger effort could be dedicated to evaluating the general fit for purpose properties with the idea developing a qualified additive that could be used by the U.S. Army in both ground and air applications.
- 9) If it is anticipated that an additive is to be used in fuel undergoing long term storage then it should be tested in the manner of expected use. In this program CN #2 was tested in combination with JP-8 additives for the SPKs and there have been significant problems with thermal stability and conductivity. Those problems could be related to the interaction with the standard additives.
- 10) The data in this program can also be taken as further proof that storing fuel with additives, other than antioxidant, is a questionable practice. Performance additives are meant to do things. To do things they have to be chemically active and giving such chemicals time, and perhaps heat, may generate adverse results.
- 11) The aviation industry has FAA guidance and support that allows placing fuel quality and compatibility ahead of commercial interest. The U.S. Army should ask the Department of Transportation to provide the same support for generating fit-for-purpose expectations for compression ignition fuels.
- 12) When doing large programs, such as this one, a statistical analysis of the program data should be a routine step. Care should be exercised, however, as short of an actual precision exercise, real world testing routinely produces poorer precision results than suggested by the method. Careful analysis of the method, comparison to known performance in programs like the ASTM ILCP and an evaluation of how the test might affect the results should still result in valuable insight.

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29. D5001 Standard Test Method for Measurement of Lubricity of Aviation Turbine Fuels by the Ball-on-Cylinder Lubricity Evaluator (BOCLE). Book of Standards Volume: 05.02. West Conshohocken, PA : ASTM International.
30. D6079 Standard Test Method for Evaluating Lubricity of Diesel Fuels by the High-Frequency Reciprocating Rig (HFRR). Book of Standards Volume: 05.02. West Conshohocken, PA : ASTM International.
31. D975 Standard Specification for Diesel Fuel Oils. Book of Standards: 5.01. West Conshohocken, PA : ASTM International.
32. D4865 Standard Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems. Book of Standards Volume: 05.02 . West Conshohocken, PA : ASTM International.

33. D3948 Standard Test Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separometer. Book of Standards Volume: 05.02. West Conshohocken, PA : ASTM International.
34. EN 15751 Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of oxidation stability by accelerated oxidation method. Brussels, Belgium : CEN-CENELEC .



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## **APPENDIX A**

### **Test Program Outline**

**WD17 Topic 2.9: Additive Effectiveness Investigations in Alternative Fuels****Test Program Outline**

1. Prepare Fuel Samples
  - a. B20 – eight (8) five gallon cans
    - i. The government spec for B20, A-A-59693A, does not require any additive
    - ii. Total fuel needed is 40 gallons
  - b. HEFA SPK – eleven (11) five gallon cans
    - i. The fuel will be additized with the normal JP-8 additives
    - ii. Total fuel needed is 55 gallons
  - c. FT SPK – eleven (11) five gallon cans
    - i. The fuel will be additized with the normal JP-8 additives
    - ii. Total fuel needed is 55 gallons
2. Prepare the Inoculums
  - a. Prepare two (2) cultures
    - i. Use dirt from soil that has had some fuel contamination
  - b. Incubate the cultures for four weeks
    - i. Monitor bio activity weekly with FQS Hyalite tester
  - c. Choose strongest culture for testing purposes
3. Prepare fuels per the SOW matrix
  - a. Additives:
    - i. Biocide: The standard stabilizer for the military is defined in MIL-S-53021A NOT 2. There is only one listed material on the QPL (qualified products list):
      1. Kathon FP 1.5 Biocide is based upon isothiazolone chemistry and technology and specifically formulated for use in distillate fuels.
    - ii. Cetane Improvers:
      1. *Alkyl Nitrate* – the primary type of Cetane Improver. This project will use the most common version, 2-ethyl hexyl nitrate (2EHN).
      2. *Peroxide* – is used where nitrous oxide (NOX) emissions are an issue. The standard peroxide is di-*tert*-butyl peroxide (DTBP).
      3. *Storage Stabilized* – a specially formulated blend of the two standard Cetane Improvers.
        - a. A thermal stability additive package is added to an admixture of the 2EHN and DTBP to improve storage stability.
        - b. Cetane Improvers have known issues with storage stability. According to paragraph X2.6.5 of ASTM D7467 (the B20 specification) “Some additives exhibit effects on fuels tested in accordance with Test Method D6468 (aka: DuPont F21)...” While D6468 is not part of the base testing (specification Table 1) it is the most likely test to be used if fuel appears to deteriorate.

## iii. JP-8 Additive Package:

1. *Fuel System Icing Inhibitor (FSII)*, di-ethylene glycol methyl ether (DIEGME) – 0.15 % by volume.
2. *Corrosion Inhibitor / Lubricity Additive* – Selected from the MIL-PRF-25017H QPL list and added at the indicated Maximum Treat Rate.
3. *Static Dissipater Additive* – Add sufficient Stadis 450 to ensure the initial blends have conductivity in the range of 150 - 600 pS/m.

## b. Blend Table

	B20 Baseline	HEFA-SPK Additized per MIL-DTL-83133 G Baseline	FT-SPK Additized per MIL-DTL-83133 G Baseline
No Additional Additives	X	X	X
Biocide	X	X	X
CN Improver #1			
max treat rate	X	X	X
1/2 max treat rate		X	X
CN Improver #2			
max treat rate	X	X	X
1/2 max treat rate		X	X
CN Improver #3			
max treat rate	X	X	X
1/2 max treat rate		X	X
Biocide + CN Improver #1 at max treat	X	X	X
Biocide + CN Improver #2 at max treat	X	X	X
Biocide + CN Improver #3 at max treat	X	X	X

## 4. Test Sample Baseline (before inoculation)

- a. B20 Testing –in accordance with ASTM D7467 (commercial B20 specification)
- b. Jet Fuel Testing – in accordance with Table I of MIL-DTL-83133G A1
  - i. Also Derived Cetane Number as specified in Table A-II
  - ii. *Note* – while the SPK samples are not fully formulated jet fuels the project is treating them as if they were.
- c. Test all fuel samples for bioactivity using FQS Hyalite system

## 5. Inoculate Samples and Place in Storage

- a. Each five gallon can will be prepared as follows:
  - i. Add 2% distilled water, by volume
  - ii. Inoculate the sample with the selected test strain

- b. Stagger the preparations to ease sampling effort
      - i. Neat and Biocide alone – Week 1, Day 1
      - ii. Cetane Improver #1 – Week 1, Day 3
      - iii. Cetane Improver #2 – Week 2, Day 1
      - iv. Cetane Improver #3 – Week 2, Day 3
    - c. Place prepared samples in 43°C walk in oven storage
- 6. Mid Term Bio Assay
  - a. After eighteen (18) weeks the samples will be evaluated for bio activity
    - i. Remove samples from the walk in oven on the same schedule as in 5b
    - ii. Allow samples to equilibrate to room temperature before opening
      - 1. Overnight should be sufficient
  - b. Sample fuel and water layers and test for bio activity with FQS Hyalite test device
- 7. Full Term Analysis
  - a. After thirty six (36) weeks the samples will be evaluated for bio activity and for specification compliance
    - i. Remove samples from the walk in oven on the same schedule as in 5b
    - ii. Allow samples to equilibrate to room temperature before opening
      - 1. Overnight should be sufficient
  - b. Sample fuel and water layers and test for bio activity with FQS Hyalite test device
  - c. Test fuel for specification properties
    - i. B20 Testing –in accordance with ASTM D7467 (commercial B20 specification)
    - ii. Jet Fuel Testing – in accordance with Table I of MIL-DTL-83133G A1
      - 1. Also Derived Cetane Number as specified in Table A-II
  - d. Place remaining samples in cold storage to stabilize for future analysis
- 8. Program Reporting
  - a. Monthly progress reports
  - b. Final report

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**APPENDIX B**  
**Chemical Hygiene Plan**

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# **Chemical Hygiene Plan**

**SwRI Project 1.08.07.12.14734.17.130**  
**George Wilson, Project Manager**

## Introduction

The U.S. Army Fuels and Lubricants Research Facility at SwRI (Army Lab) routinely tests commercial and military jet fuel, diesel fuel and variations thereof. Additionally the Army Lab routinely works with the standard additives associated with these fuels. As a result, the Army Lab is used to working with these materials in a safe fashion. This program will be dealing with additive materials not commonly used in these facilities. This requires a review of the health and safety issues surrounding these materials.

## The Additives

1. Biocide
  - a. KATHON FP1.5 Biocide (CL12-3352)
2. Cetane Improvers
  - a. CN #1: Innospec CI-0801 (CL12-3365)
  - b. CN #2: Innospec CI-0808 (CL12-3361)
  - c. CN #3: Innospec CI-0802 (CL12-3366)

MSDS Information: The MSDS Sheets for these products are attached. Take time to review the MSDS for each additive before handling. These chemicals have hazards not typical of products normally used in the Army Lab. Following is a brief discussion of each additive. This is not a substitute for reviewing the MSDS.

### KATHON FP1.5 Biocide (CL12-3352)

Biocide – Poison! Take special care when using this material. It is in a plastic bottle. Keep this bottle in secondary containment when in use and storage (after breaking the seal). Put the bottle in a glass beaker to keep it from being tipped over in the secondary containment. Cap and seal the bottle any time it is not in immediate use.

Use goggles and a face shield when handling this material.

Use butyl or nitrile gloves at all times when handling this material. Place a note so stating on the secondary containment. When handling this material do not touch any other part of your body or clothes with your gloved hand. Remove and discard gloves into hazardous waste when leaving the work area. Do not reuse gloves.

Wear a chemical resistant apron.

If you get this material on you it will require immediate medical attention. If it is on your clothes they will have to be removed immediately and cleaned before reuse. You may not clean them at home. Contaminated leather goods will have to be discarded.

All disposable materials used in handling this product should be treated as hazardous waste. This included gloves, disposable pipettes, paper towels, etc.

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**CN #1: Innospec CI-0801 (CL12-3365)**

2-ethyl hexyl nitrate (2EHN). This is the most common Cetane improver in use today. It is not as toxic as the biocide above but it is a potential hazard. You should still use goggles but a splash shield is not required.

This material has more solvency than a regular fuel. Nitrile gloves should be discarded after one hour of use. (The MSDS says use time is 1 to 4 hours but we will be conservative.)

Note this material decomposes above 100°C. Avoid spilling any of it on hot surfaces.

All disposable materials need to go into hazardous waste.

**CN #2: Innospec CI-0808 (CL12-3361)**

This is a peroxide based Cetane improver. The active ingredient is di-tert-butyl peroxide, DTB-Peroxide. This is one of the most stable of the organic peroxides but it still has to be treated with care. Because of potential eye issues, this material needs to be handled using a splash guard in addition to the goggles.

This MSDS recommends Viton gloves for handling, however, a literature search indicated that nitrile is sufficient. Use the same 1 hour, no reuse glove limit as indicated for the 2EHN.

This material also decomposed above 100°C. In addition, it has a low flash point and is flammable so in addition to hot surfaces this product needs to be kept away from ignition sources.

Peroxides are very reactive and often used as catalysts for polymerization. Inadvertent contact with reactive chemicals could start an uncontrolled reaction. Handle used laboratory equipment carefully and clean with neutral solvent to dilute peroxide concentration.

All disposable materials need to go into hazardous waste.

**CN #3: Innospec CI-0802 (CL12-3366)**



This is also a 2EHN Cetane improver but with added compounds to improve storage stability. Handle it in the same fashion as CN #2



# MSDS Sheets Following:

## 1. Biocide

### a. KATHON FP1.5 Biocide


 <b>Fuel Quality Services, Inc.</b>	 <b>Certificate No. TRC 00346</b>																					
<small>P.O. Box 1380, Flowery Branch, GA. 30542-0023 • Phone: 800-827-9790 or 770-967-9790 • Fax: 770-967-9982 Website: <a href="http://www.fqsinc.com">www.fqsinc.com</a></small>																						
<b>Material Safety Data Sheet</b>																						
<b>1 – PRODUCT AND COMPANY IDENTIFICATION</b>																						
<b>Kathon™ FP 1.5 Biocide</b>																						
<p>MSDS: November 29, 2006</p> <table border="0" style="width: 100%;"><tr><td style="width: 50%; vertical-align: top;"><p><b>Company Identification:</b> Rohm &amp; Haas Company 100 Independence Mall West Philadelphia, PA 19106-2399 USA</p></td><td style="width: 50%; vertical-align: top;"><p><b>Emergency Telephone Numbers:</b> Spill Emergency: 215-592-3000 Health Emergency: 215-592-3000 CHEMTREC: 800-424-9300</p></td></tr></table> <p>For non-emergency information contact: 770-967-9790</p> <p><b>Distributed By:</b> Fuel Quality Services, Inc. P.O. Box 1380 Flowery Branch, GA 30542 USA Email: <a href="mailto:technical@fqsinc.com">technical@fqsinc.com</a> Website: <a href="http://www.fqsinc.com">www.fqsinc.com</a></p> <p style="text-align: right;">Telephone: 770-967-9790</p>		<p><b>Company Identification:</b> Rohm &amp; Haas Company 100 Independence Mall West Philadelphia, PA 19106-2399 USA</p>	<p><b>Emergency Telephone Numbers:</b> Spill Emergency: 215-592-3000 Health Emergency: 215-592-3000 CHEMTREC: 800-424-9300</p>																			
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<b>2 - COMPOSITION / INFORMATION ON INGREDIENTS</b>																						
<table border="0" style="width: 100%;"><thead><tr><th style="text-align: left;"><u>Component</u></th><th style="text-align: left;"><u>CAS No</u></th><th style="text-align: left;"><u>Concentration</u></th></tr></thead><tbody><tr><td>5-Chloro-2-methyl-4-isothiazolin-3-one</td><td>26172-55-4</td><td>1.0 - 1.3%</td></tr><tr><td>2-Methyl-4-isothiazolin-3-one</td><td>2682-20-4</td><td>0.3 - 0.4%</td></tr><tr><td>Magnesium nitrate</td><td>10377-60-3</td><td>1.7 - 1.8%</td></tr><tr><td>Magnesium Chloride</td><td>7786-30-3</td><td>0.9 - 1.0%</td></tr><tr><td>Water</td><td>7732-18-5</td><td>5.0 - 6.0%</td></tr><tr><td>Dipropylene glycol (Mixed isomers)</td><td>25265-71-8</td><td>88.0 - 90.0%</td></tr></tbody></table>		<u>Component</u>	<u>CAS No</u>	<u>Concentration</u>	5-Chloro-2-methyl-4-isothiazolin-3-one	26172-55-4	1.0 - 1.3%	2-Methyl-4-isothiazolin-3-one	2682-20-4	0.3 - 0.4%	Magnesium nitrate	10377-60-3	1.7 - 1.8%	Magnesium Chloride	7786-30-3	0.9 - 1.0%	Water	7732-18-5	5.0 - 6.0%	Dipropylene glycol (Mixed isomers)	25265-71-8	88.0 - 90.0%
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<small>Kathon™ FP 1.5</small>	<small>Page 1 of 8</small>	<small>FQS-105-13 01/08</small>																				

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## 2. Cetane Improvers

### a. Innospec CI-0801



# CI-0801

## Material Safety Data Sheet

### 1. Product and company identification

**Common name** : CI-0801

**Synonym** : Nitric acid, 2-ethylhexyl ester (CAS); 3-Nitroxymethyl-heptane (IUPAC) ; Alkyl (C7-C9) nitrates, all isomers

**Trade name** : 2EHN

**CAS number** : 27247-98-7

**Product type** : Liquid.

**Material uses** : Petrochemical industry: Petrochemicals. Fuel additive.

**Internal code** : 10400

**Supplier** : Innospec Fuel Specialties LLC  
North American Headquarters  
8375 South Willow Street  
Littleton  
Colorado 80124  
USA

**Information contact** : 1-800-441-9547

**Emergency telephone number**

In USA, Canada and North America, 24 hour / 7 day emergency response for Innospec products is provided by the CHEMTREC (R) Emergency Call Center based in the USA  
toll-free telephone numbers USA : 800 424 9300 Canada, Puerto Rico, Virgin Islands : +1 800 424 9300  
In case of difficulty using the toll-free number, or for ships at sea, please call +1 703 527 3887

In Europe, Middle East, Africa, Asia Pacific and South America  
24 hour / 7 day emergency response for Innospec products is provided by the NCEC CARECHEM 24 global network

The main regional centres are listed here in Section 1.  
Other local contact numbers for specific language support in Asia Pacific are listed in Section 16

Country information	Emergency telephone number	Location
Europe ( all countries, all languages )	+44 (0) 1235 239 670	London, UK
Middle East, Africa ( Arabic, French, English )	+44 (0) 1235 239 671	Lebanon
Middle East, Africa ( French, Portuguese, English )	+44 (0) 1235 239 670	London UK
Asia Pacific ( all countries except China )	+65 3158 1074	Singapore
China	+86 10 5100 3039	Beijing China
South America ( all countries )	+1 215 207 0081	Philadelphia USA

### 2. Hazards identification

**Physical state** : Liquid.

**Odor** : Characteristic. Pungent. [Strong]

**OSHA/HCS status** : This material is considered hazardous by the OSHA Hazard Communication Standard (29 CFR 1910.1200).

**Emergency overview** : WARNING!


This Material Safety Data Sheet conforms to the requirements of ANSI Z400.1.

Date of issue : April 11, 2011

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## b. Innospec CI-0808



### CI-0808

## Material Safety Data Sheet

#### 1. Product and company identification

<b>Common name</b>	: CI-0808
<b>Material uses</b>	: Petrochemical industry: Fuel additive.
<b>Internal code</b>	: IFS0138
<b>Supplier</b>	: Innospec Fuel Specialties LLC North American Headquarters 8375 South Willow Street Littleton Colorado 80124 USA
<b>Information contact</b>	: 1-800-441-9547
<b>In case of emergency</b>	: 1-800-424-9300 (Chemtrec)

#### 2. Hazards identification

<b>Physical state</b>	: Liquid.
<b>Odor</b>	: Mild [Strong]
<b>OSHA/HCS status</b>	: This material is considered hazardous by the OSHA Hazard Communication Standard (29 CFR 1910.1200).
<b>Emergency overview</b>	: WARNING!  FLAMMABLE LIQUID AND VAPOR. CAUSES EYE IRRITATION. MAY CAUSE RESPIRATORY TRACT AND SKIN IRRITATION. CONTAINS MATERIAL THAT CAN CAUSE TARGET ORGAN DAMAGE. POSSIBLE CANCER HAZARD - CONTAINS MATERIAL WHICH MAY CAUSE CANCER, BASED ON ANIMAL DATA.  Flammable liquid. Severely irritating to eyes. Slightly irritating to the skin and respiratory system. Keep away from heat, sparks and flame. Avoid exposure - obtain special instructions before use. Do not breathe vapor or mist. Do not get in eyes. Avoid contact with skin and clothing. Contains material that can cause target organ damage. Contains material which may cause cancer, based on animal data. Risk of cancer depends on duration and level of exposure. Use only with adequate ventilation. Keep container tightly closed and sealed until ready for use. Wash thoroughly after handling.

Potential acute health effects

Inhalation	: Slightly irritating to the respiratory system.
Ingestion	: No known significant effects or critical hazards.
Skin	: Slightly irritating to the skin.
Eyes	: Severely irritating to eyes. Risk of serious damage to eyes.

Potential chronic health effects

Chronic effects	: Contains material that can cause target organ damage.
Carcinogenicity	: Contains material which may cause cancer, based on animal data. Risk of cancer depends on duration and level of exposure.
Mutagenicity	: No known significant effects or critical hazards.
Teratogenicity	: No known significant effects or critical hazards.
Developmental effects	: No known significant effects or critical hazards.
Fertility effects	: No known significant effects or critical hazards.
Target organs	: Contains material which causes damage to the following organs: upper respiratory tract, skin, eye, lens or cornea. Contains material which may cause damage to the following organs: kidneys.

Over-exposure signs/symptoms

This Material Safety Data Sheet conforms to the requirements of ANSI Z400.1.

Date of issue : January 28, 2009 Page: 1/8

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## c. Innospec CI-0802



### CI-0802 ej1 Material Safety Data Sheet

#### 1 Company Identification

Innospec Fuel Specialties  
8375 S. Willow Street  
Littleton, CO 80124

Product information 1-800-441-9547  
In Case of Emergency  
Call Chemtrec 1-800-424-9300

#### 2 Composition / Ingredient Information

<u>Material</u>	<u>CAS Number</u>	<u>%</u>
2-Ethylhexyl Nitrate.....	27247-96-7.....	90-100
2-Ethylhexyl Alcohol.....	104-76-7.....	<2
Detergent.....		<10
Heavy Aromatic Naphtha.....	64742-94-5.....	<5
*(Naphthalene).....	91-20-3.....	(<0.5)

\*Disclosure as a toxic chemical is required under Section 313 of Title III of the Superfund Amendments and Reauthorization Act of 1986 and 40 CFR part 372.

#### 3 Hazardous Identification

##### Potential Health Effects

Skin contact with Detergent may cause skin sensitization upon extended contact. The compound may cause skin sensitization in susceptible individuals. Eye contact may cause eye irritation with discomfort, tearing, or blurring of vision. Inhalation may initially include irritation of the upper respiratory passages with coughing and discomfort. Individuals with preexisting diseases of the central nervous system may have increased susceptibility to the toxicity of excessive exposures to Detergent.

Inhalation or ingestion of Heavy Aromatic Naphtha may cause central nervous system depression with anesthetic effects, such as dizziness, headache, confusion, incoordination and loss of consciousness. Higher exposures may result in fatality from gross overexposure. Ingestion may cause gastrointestinal irritation. Aspiration hazard! Small amounts aspirated into the lungs during ingestion or vomiting may cause lung injury, possibly leading to death. Symptoms of aspiration into the lungs include coughing, gasping, choking, shortness of breath, bluish discolored skin, rapid breathing and heart rate. Chemical pneumonitis from aspiration may result in fever. Pulmonary edema or bleeding, drowsiness, confusion, coma and seizures may occur in more serious cases. Symptoms may develop immediately or as late as 24 hours after the exposure, depending on how much chemical entered the lungs.

Minute amounts of petroleum hydrocarbons aspirated into the lungs during ingestion or vomiting may cause mild to severe pulmonary injury and possible death.

CI-0802 ej1  
Revised 3/12/07  
Revision 1

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Innospec Fuel Specialties

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**APPENDIX C**  
**Test Results**

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In the data tables that follow, some of the interesting results are annotated. The meaning of that annotation is described in Table C-1 below.

**Table C-1. Table Annotations**

Annotation	Definition
<b>BAD</b>	Results do not meet commonly accepted standards for this value
<b>INTERESTING</b>	Results that are within acceptable limits but, by comparison, indicate additive effect

In this annex the cumulative data for the program is presented. The data is arranged in the following order:

- 1) FT SPK Blend Data - Start of Test (SOT), Table C-
- 2) FT SPK Blend Data - End of Test (EOT), Table C-
- 3) HEFA SPK Blend Data - Start of Test (SOT), Table C-
- 4) HEFA SPK Blend Data - End of Test (EOT), Table C-5
- 5) B20 Blend Data - Start of Test (SOT), Table C-
- 6) B20 Blend Data - End of Test (EOT), Table C-

Table C-2. FT SPK Blend Data – Start of Test (SOT)

Additives		FT-1	FT-2	FT-3	FT-4	FT-5	FT-6	FT-7	FT-8	FT-9	FT-10	FT-11
Biocide - Kathon FP 1.5		-	100 ppm	-	-	-	-	-	-	100 ppm	100 ppm	100 ppm
CN #1 - 2 ethyl hexyl nitrate (2EHN)		-	-	0.80 vol %	0.40 vol %	-	-	-	-	0.80 vol %	-	-
CN #2 - 2EHN, with storage stability additives		-	-	-	-	0.50 vol %	0.25 vol %	-	-	-	0.50 vol %	-
CN #3 - Di Tert Butyl Peroxide (DTBP)		-	-	-	-	-	-	0.50 vol %	0.25 vol %	-	-	0.50 vol %
SwRI Sample ID		CL12-3464 FT-1, CAF- 7051, 2/15/12	CL12-3465 FT-2, CAF- 7051, 2/15/12	CL12-3466 FT-3, CAF- 7051, 2/15/12	CL12-3467 FT-4, CAF- 7051, 2/15/12	CL12-3468 FT-5, CAF- 7051, 2/15/12	CL12-3535 FT-11, CAF- 7051, 2/15/12	CL12-3470 FT-7, CAF- 7051, 2/15/12	CL12-3471 FT-8, CAF- 7051, 2/15/12	CL12-3472 FT-9, CAF- 7051, 2/15/12	CL12-3473 FT-10, CAF- 7051, 2/15/12	CL12-3474 FT-11, CAF- 7051, 2/15/12
Description												
D1319	Aromatic Content											
	Aromatics	%	1.3	1.2	1.8	1.4	1.5	0.5	1.4	1.2	1.8	1.5
	Olefins	%	0.6	0.8	0.8	0.6	0.7	0.5	0.4	0.4	0.6	0.5
	Saturates	%	98.1	98	97.4	98	97.8	99.0	98.2	98.4	97.6	98.2
D2624	Electrical Conductivity	pS/m	18	7	8	4	342	490	9	10	4	514
		°C	23.1	23.2	23.2	23.2	23.2	21.6	22.9	23.1	23.1	23.3
D3241	Jet Fuel Thermal Stability											
	Test Temperature	°C	275	275	275	275	275	275	275	275	275	275
	ASTM Code		<2	<2	<2	<2	4AP	1	<2	<2	>4AP	<2
	Maximum mm Hg	mm Hg	0.1	0	0	0	0	0.5	0.1	0	0.1	0.1
D3242	Acidity (Aviation)	mg KOH/g	0.016	0.02	0.018	0.018	0.018	0.014	0.019	0.011	0.012	0.012
D3701	Hydrogen by Nuclear Magnetic Resonance	mass %	15.51	15.3	15.29	15.38	15.45	15.45	15.41	15.5	15.42	15.39
D4052	Density at 15°C	g/mL	0.7423	0.7423	0.744	0.7432	0.7433	0.7368	0.7426	0.7425	0.7441	0.7435
D445	Viscosity - Kinematic at -20°C	cSt	2.59	2.59	2.6	2.59	2.59	2.48	2.59	2.57	2.58	2.54
D4809	Heat of Combustion – Net	MJ/kg	43.5	43.7	43.6	43.8	43.8	43.8	43.5	44.0	43.6	43.7
D5291	Carbon/Hydrogen											
	Carbon	mass %	84.32	84.36	84.46	84.35	84.35	84.17	84.43	84.75	84.25	84.65
	Hydrogen	mass %	15.37	15.31	15.38	15.45	15.38	15.51	15.3	15.54	15.46	15.5
	Carbon + Hydrogen	mass %	99.69	99.67	99.84	99.8	99.73	99.68	99.73	100.29	99.71	100.12
D5453	Sulfur – UV	ppm	1.5	1.9	2.9	2	1.9	2.4	1.5	1.1	3.2	3
D6890	IQT											
	Derived Cetane Number		59	58.95	93.04	82.6	81.9	78.14	73.81	69.29	92.55	84.26
	Ignition Delay	ms	3.421	3.425	2.42	2.608	2.623	2.71	2.823	2.963	2.428	2.574
D3948	MSEP		95	97	95	96	45	32	88	94	95	52
D86	Distillation											
	Initial Boiling Point	°C	157.3	157.3	157.6	156.9	156.7	156.7	156.3	156.7	157.3	156.8
	5% recovery	°C	162.6	162.4	162.7	162.2	162.4	161.6	162.2	162.1	162.2	162.4
	10% recovery	°C	163.2	163	163.1	163	163.3	162	162.7	163	163.2	163.4
	15% recovery	°C	163.5	163.6	163.5	163.7	163.8	162.5	163.4	163.5	163.9	163.8
	20% recovery	°C	164.5	164.2	164.1	164.6	164.7	163.1	164.6	164.5	164.5	164.5
	30% recovery	°C	166.6	166.7	166.8	166.6	166.7	165.2	166.5	166.5	166.5	166.6
	40% recovery	°C	168.6	168.5	168.7	168.7	168.9	167.1	168.8	168.7	168.8	168.8
	50% recovery	°C	170.8	171.1	171.1	171.2	171.2	169.1	171.3	171.1	171.1	171
	60% recovery	°C	173.8	173.8	173.7	173.8	173.8	171.6	174	173.9	173.8	174
	70% recovery	°C	177.3	177.2	177.2	177.5	177.5	174.6	177.6	177.6	177.4	177.2
	80% recovery	°C	182.4	182.3	182.3	182.4	182.5	178.6	182.4	182.5	182.3	182.5
	90% recovery	°C	190.5	190.4	190.5	190.7	190.8	184.9	190.6	190.4	190.4	190.7
	95% recovery	°C	199.7	199.4	199.7	200.3	200.3	190.6	199.7	199.4	198.7	199.1
	Final Boiling Point	°C	217.8	216.7	214.2	216.1	215.2	205.4	217.3	216.9	213.7	215.7
	Residue	mL	1.2	1.2	1.4	1.3	1.6	1.1	1.3	1.2	1.3	1.3
	Loss	mL	0.6	0.5	0.4	0.6	0.6	0.4	0.4	0.4	0.3	0.2
	T90-T10	°C	27.3	27.4	27.4	27.7	27.5	22.9	27.9	27.4	27.2	27.3
D5972	Freeze Point	°C	-53.5	-55.1	-55.1	-54.5	-54.5	-53.8	-54.5	-54.6	-54.5	-55.1
D93	Flash Point (Pensky-Martin)	°C	43.0	43.0	43.0	43.0	43.0	42.0	42.0	42.0	43.0	42.0
D156	Saybolt color		26	27	27	27	24	20	28	27	26	22
D4294	Sulfur-Energy Dispersive	wt.%	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
D3227	Sulfur-Mercaptan	mass %	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003
D3338	Specific Energy	MJ/Kg	44.2	44.2	44.1	44.1	44.1	44.2	44.2	44.2	44.1	44.1
D1322	Smoke Point	mm	25	25	24	24	24	24.5	25.5	22	24	25
D976	Cetane Index		56	56.2	55.4	55.8	55.8	57.8	56.2	56.1	55.3	55.6
D130	Copper Strip Corrosion											
	Code	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A
	°C	100	100	100	100	100	100	100	100	100	100	100
	Hours	2	2	2	2	2	2	2	2	2	2	2
D381	Existent Gum	g/100ml	16	18	16	19	31	22	18	19	20	47
D5006	Fuel System Icing Inhibitor Content	vol %	0.16	0.18	0.16	0.17	0.16	0.07	0.16	0.17	N/A	0.19
SWRI	Hy-LITE		21	16	110	13	8	10	56	58	160	15
D5001	BOCLE	mm	0.48	0.49	0.47	0.51	0.45	0.56	0.51	0.51	0.48	0.44

Table C-3. FT SPK Blend Data – End of Test (EOT)

Additives			FT-1	FT-2	FT-3	FT-4	FT-5	FT-6	FT-7	FT-8	FT-9	FT-10	FT-11
Biocide - Kathon FP 1.5			-	100 ppm	-	-	-	-	-	-	100 ppm	100 ppm	100 ppm
CN #1 - 2 ethyl hexyl nitrate (2EHN)			-	-	0.80 vol %	0.40 vol %	-	-	-	-	0.80 vol %	-	-
CN #2 - 2EHN, with storage stability additives			-	-	-	-	0.50 vol %	0.25 vol %	-	-	-	0.50 vol %	-
CN #3 - Di Tert Butyl Peroxide (DTBP)			-	-	-	-	-	-	0.50 vol %	0.25 vol %	-	-	0.50 vol %
	SwRI Sample ID		CL12-3464	CL12-3465	CL12-3466	CL12-3467	CL12-3468	CL12-3735	CL12-3470	CL12-3471	CL12-3472	CL12-3473	CL12-3474
	Description		FT-1, CAF-7051, 2/15/12	FT-2, CAF-7051, 2/15/12	FT-3, CAF-7051, 2/15/12	FT-4, CAF-7051, 2/15/12	FT-5, CAF-7051, 2/15/12	FT-11, CAF-7051, 2/15/12	FT-7, CAF-7051, 2/15/12	FT-8, CAF-7051, 2/15/12	FT-9, CAF-7051, 2/15/12	FT-10, CAF-7051, 2/15/12	FT-11, CAF-7051, 2/15/12
D1319	Aromatic Content												
	Aromatics	%	0.9	0.9	1.5	1.1	1.2	0.2	1	1	1.5	1.2	1
	Olefins	%	0.7	0.5	0.3	0.4	0.4	0.2	0.5	0.3	0.4	0.4	0.4
	Saturates	%	98.4	98.6	98.2	98.5	98.4	99.6	98.5	98.7	98.1	98.4	98.6
D2624	Electrical Conductivity	pS/m	89	59	41	18	3090	339	22	32	19	4280	23
		°C	18.7	20.2	19.5	20.4	21.2	21.5	19.1	21	21	19	21.3
D3241	Jet Fuel Thermal Stability												
	Test Temperature	°C	275	275	275	275	275	275	275	275	275	275	275
	ASTM Code		<1	<1	<1	<1	2	<1	<1	<1	<1	<2	<1
	Maximum mm Hg	mm Hg	0	0	0.1	0	0.1	0	0	0	0	0	0.1
D3242	Acidity (Aviation)	mg KOH/g	0.01	0.008	0.008	0.007	0.01	0.011	0.008	0.006	0.008	0.007	0.006
D3701	Hydrogen by Nuclear Magnetic Resonance	mass %	15.31	15.33	15.47	15.32	15.45	15.69	15.54	15.56	15.48	15.34	15.49
D4052	Density at 15°C	g/mL	0.7421	0.7420	0.7438	0.7429	0.7429	0.7368	0.7423	0.7429	0.7439	0.7432	0.7426
D445	Viscosity - Kinematic at -20°C	cSt	2.60	2.60	2.59	2.59	2.61	2.49	2.60	2.58	2.59	2.59	2.60
D4809	Heat of Combustion – Net	MJ/kg	43.4	43.9	44.3	43.8	44.0	44.1	44.0	44.3	43.8	43.9	44.1
D5291	Carbon/Hydrogen												
	Carbon	mass %	84.47	84.52	84.33	84.52	84.63	84.28	84.50	84.85	84.78	84.53	84.74
	Hydrogen	mass %	15.38	15.48	15.44	15.42	15.47	15.52	15.43	14.46	15.45	15.43	15.41
	Carbon + Hydrogen	mass %	99.85	100.00	99.77	99.94	100.10	99.80	99.93	99.31	100.23	99.96	100.15
D5453	Sulfur – UV	ppm	1.1	1.2	3	2.2	2.1	2.5	1.3	0.7	2.9	2.3	1.6
D6890	IQT												
	Derived Cetane Number		57.01	57.04	88.94	80.3	80.12	64.64	72.29	67.58	87.91	80.75	72.05
	Ignition Delay	ms	3.551	3.549	2.487	2.659	2.663	3.101	2.868	3.022	2.505	2.649	2.875
D3948	MSEP		98	94	94	94	0	0	98	97	77	0	98
D86	Distillation												
	Initial Boiling Point	°C	157.8	157.2	157.4	157.4	157.3	156.6	157	157.2	157.6	157.6	156.4
	5% recovery	°C	162.6	162.6	162.5	162.1	162.2	161.3	162	162.2	162	162.4	161.9
	10% recovery	°C	163.2	163	162.9	163	162.9	161.9	162.5	162.9	162.9	163.2	162.7
	15% recovery	°C	163.5	163.6	163.5	163.6	163.6	162.5	163.2	163.4	163.6	160.1	163.3
	20% recovery	°C	164.4	164.2	164.2	164	164.3	163.1	164	164.1	164.1	162.7	164
	30% recovery	°C	166.5	166.8	166.5	166.7	166.6	165.3	166.5	166.6	166.7	166.3	166.7
	40% recovery	°C	168.5	168.5	168.5	168.5	168.5	166.9	168.5	168.5	168.3	168.5	168.7
	50% recovery	°C	170.9	170.9	170.7	170.9	170.9	169	171	170.8	170.8	170.8	171
	60% recovery	°C	173.5	173.7	173.6	173.5	173.5	171.3	173.8	173.7	173.5	173.6	173.7
	70% recovery	°C	177.1	177	176.9	177	177.1	174.4	177.3	177.2	176.9	177.1	177.2
	80% recovery	°C	181.9	181.9	182.1	182	182.1	178.5	182.3	182	181.9	182	182.2
	90% recovery	°C	189.5	190	190.1	190.1	190.2	184.6	190.4	190	189.9	189.8	190.4
	95% recovery	°C	197.6	198.6	199	199	199.5	190.4	200.3	198.7	199.2	198.9	199.7
	Final Boiling Point	°C	217.3	216.7	213.2	215.6	215.3	204.5	208.9	216.4	214.3	214.9	218.2
	Residue	mL	1.1	1.4	1.5	1.4	1.3	1.2	1.5	1.3	1.4	1.3	1.4
	Loss	mL	0.1	0.1	0.2	0.1	0.3	0.3	0.3	0.2	0.2	0.2	0.2
	T90-T10	°C											
D5972	Freeze Point	°C	-55	-56	-55	-57.1	-54.9	-53.6	-56.2	-56.3	-56.5	-54.6	-58.3
D93	Flash Point (Pensky-Martin)	°C	42.5	42.5	43.5	43.5	42.5	40.5	41.5	42.0	43.0	41.5	41.5
D156	Saybolt color		26	26	23	23	16	17	19	22	22	17	19
D4294	Sulfur-Energy Dispersive	wt.%	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
D3227	Sulfur-Mercaptan	mass %	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003
D3338	Specific Energy	MJ/Kg	44.164	44.165	44.128	44.149	44.147	44.238	44.159	44.151	44.126	44.143	44.155
D1322	Smoke Point	mm	48	48	47	46	45	47	48	49	47	46	45
D976	Cetane Index		56.2	56.2	55.2	55.8	55.8	57.7	56.1	55.7	55.2	55.6	56
D130	Copper Strip Corrosion												
	Code	1A	1A	1A	1A	1A	1A	1A	1B	1B	1A	1A	1A
	°C	100	100	100	100	100	100	100	100	100	100	100	100
	Hours	2	2	2	2	2	2	2	2	2	2	2	2
D381	Existent Gum	g/100ml	1	1	<0.5mg/100mL	1	18	1	<0.5mg/100mL	<0.5mg/100mL	<0.5mg/100mL	22	1
D5006	Fuel System Icing Inhibitor Content	vol %	0.00	0.00	0.00	0.00	0.01	0.01	0.00	0.00	0.00	0.01	0.00
SWRI	Hy-LITE (fuel)		20	11	13	11	12	17	12	10	20	17	13
SWRI	Hy-LITE (water)		20	14	30	24	13	17	18	11	15	37	8
D5001	BOCLE	mm	0.53	0.6	0.5	0.55	0.57	0.58	0.56	0.56	0.55	0.6	0.59



**Table C-4. HEFA SPK Blend Data – Start (SOT)**

Additives			HRJ-1	HRJ-2	HRJ-3	HRJ-4	HRJ-5	HRJ-6	HRJ-7	HRJ-8	HRJ-9	HRJ-10	HRJ-11
Biocide - Kathon FP 1.5			-	100 ppm	-	-	-	-	-	-	100 ppm	100 ppm	100 ppm
CN #1 - 2 ethyl hexyl nitrate (2EHN)			-	-	0.80 vol %	0.40 vol %	-	-	-	-	0.80 vol %	-	-
CN #2 - 2EHN, with storage stability additives			-	-	-	-	0.50 vol %	0.25 vol %	-	-	-	0.50 vol %	-
CN #3 - Di Tert Butyl Peroxide (DTBP)			-	-	-	-	-	-	0.50 vol %	0.25 vol %	-	-	0.50 vol %
SwRI Sample ID			CL12-3453	CL12-3454	CL12-3455	CL12-3456	CL12-3457	CL12-3458	CL12-3459	CL12-3460	CL12-3461	CL12-3462	CL12-3463
Description			HRJ-1, CAF-7815, 2/15/12	HRJ-2, CAF-7815, 2/15/12	HRJ-3, CAF-7815, 2/15/12	HRJ-4, CAF-7815, 2/15/12	HRJ-5, CAF-7815, 2/15/12	HRJ-6, CAF-7815, 2/15/12	HRJ-7, CAF-7815, 2/15/12	HRJ-8, CAF-7815, 2/15/12	HRJ-9, CAF-7815, 2/15/12	HRJ-10, CAF-7815, 2/15/12	HRJ-11, CAF-7815, 2/15/12
D1319	Aromatic Content												
	Aromatics	%	0.4	0.5	0.6	0.4	0.4	0.4	0.3	0.2	0.7	0.4	0.5
	Olefins	%	0.5	0.7	0.4	0.3	0.4	0.4	0.3	0.2	0.6	0.5	0.4
	Saturates	%	99.1	98.8	99	99.3	99.2	99.2	99.4	99.6	98.7	99.1	99.1
D2624	Electrical Conductivity	pS/m	9	6	5	5	246	138	6	9	5	334	7
		°C	24.0	24.0	24.0	24.0	24.0	24.0	24.0	24.0	22.8	23.0	23.0
D3241	Jet Fuel Thermal Stability												
	Test Temperature	°C	275	275	275	275	275	275	275	275	275	275	275
	ASTM Code		<2	<2	<2	<2	4AP	>4P	<2	<2	<2	>4AP	>4P
	Maximum mm Hg	mm Hg	0.1	0.1	0	0	0.1	0	0	0.1	0	0.1	0
D3242	Acidity (Aviation)	mg KOH/g	0.01	0.014	0.017	0.02	0.025	0.022	0.011	0.012	0.015	0.019	0.015
D3701	Hydrogen by Nuclear Magnetic Resonance	mass %	15.21	15.15	15.25	15.3	15.19	15.35	15.23	15.22	15.32	15.24	15.33
D4052	Density at 15°C	g/mL	0.762	0.762	0.762	0.7628	0.7629	0.7624	0.7622	0.762	0.7636	0.7631	0.7623
D445	Viscosity - Kinematic at -20°C	cSt	5.91	5.9	5.81	5.77	5.82	5.84	5.8	5.81	5.82	5.86	5.78
D4809	Heat of Combustion – Net	MJ/kg	43.6	43.7	43.7	43.4	43.6	43.5	43.8	43.8	43.5	43.6	43.8
D5291	Carbon/Hydrogen												
	Carbon	mass %	84.59	84.59	84.24	84.47	84.4	84.56	84.46	84.43	84.41	84.49	84.45
	Hydrogen	mass %	15.18	15.12	15.41	15.27	15.36	15.14	15.27	15.24	15.42	15.37	15.29
	Carbon + Hydrogen	mass %	99.77	99.71	99.65	99.74	99.76	99.7	99.73	99.67	99.83	99.86	99.74
D5453	Sulfur – UV	ppm	1.2	1.1	2.9	1.8	2	1.3	0.8	0.9	3	2.3	1.1
D6890	IQT												
	Derived Cetane Number		61	61.05	93.64	84.82	84.6	78.25	74.1	70.22	93.64	85.63	74.61
	Ignition Delay	ms	3.3	3.297	2.411	2.563	2.564	2.707	2.815	2.932	2.406	2.547	2.801
D3948	MSEP		88	76	70	89	0	0	76	80	83	3	58
D86	Distillation												
	Initial Boiling Point	°C	148	148.8	149.3	148.6	149.6	149.9	148.9	149.1	150.1	149.9	148.5
	5% recovery	°C	162	162.3	162.1	162.9	161.8	162.9	161.9	161.5	161.7	161.5	161.6
	10% recovery	°C	166.9	166.4	165.1	166.7	166.4	166.7	164.7	166.2	165.2	165.8	165.7
	15% recovery	°C	172.6	172	171.2	172.1	172.7	173	171.7	171.9	171.8	171.7	172.4
	20% recovery	°C	178.6	178	177	177.9	178.1	178.5	177.4	178.1	176.8	177.6	178.6
	30% recovery	°C	192.5	192.4	190.8	192.5	192.5	193.5	193.4	193.4	191.3	192.1	193.6
	40% recovery	°C	208.2	208.5	208.2	209.4	210	210.6	210.4	210.3	209.1	209.4	210.1
	50% recovery	°C	225.8	225.7	226	226.3	227.4	227.7	227.1	227.1	226.3	226.6	227.2
	60% recovery	°C	241.4	241.7	241.5	242	243	243	242.8	243.2	241.9	242.3	242.1
	70% recovery	°C	255.2	255.8	255.5	256.1	256.7	256.8	256.6	256.7	256.1	256.1	256.3
	80% recovery	°C	266.7	266.8	266.8	267.1	267.5	267.5	267.3	267.6	267.1	267.1	267
	90% recovery	°C	274.8	274.7	275	275.2	275.2	275.4	275.5	275.4	275.3	275.3	275.2
	95% recovery	°C	278.4	278.5	278.9	279	279.2	279.2	279.2	279.3	279.3	278.9	279
	Final Boiling Point	°C	281.5	281.9	282.2	282.4	281.5	281.7	284.3	283.1	282.6	281.7	283.5
	Residue	mL	1.3	1.3	1.4	1.4	1.4	1.4	1.3	1.3	1.4	1.4	1.3
	Loss	mL	0.3	0.3	0.3	0.5	0.8	0.6	0.6	0.7	0.6	0.4	0.5
	T90-T10	°C	107.9	108.3	109.9	108.5	108.8	108.7	110.8	109.3	110.2	109.5	109.5
D5972	Freeze Point	°C	-55.5	-53.6	-55.8	-55.7	-55.6	-55.7	-53.9	-53.6	-53.1	-55	-55.5
D93	Flash Point (Pensky-Martin)	°C	43.0	43.0	43.0	43.0	43.0	42.0	41.0	41.0	46.0	43.0	45.0
D156	Saybolt color		19	19	20	20	19	19	20	21	20	17	21
D4294	Sulfur-Energy Dispersive	wt.%	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
D3227	Sulfur-Mercaptan	mass %	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003
D3338	Specific Energy	MJ/Kg	44.1	44.1	44.1	44.1	44.1	44.1	44.1	44.1	44.1	44.1	44.1
D1322	Smoke Point	mm	25	24.5	25.5	24.5	25.5	25	22	25	25	25.5	25.5
D976	Cetane Index		70.6	70.5	69.9	70.4	70.7	71	70.9	71	70	70.3	70.9
D130	Copper Strip Corrosion												
	Code	1B	1B	1B	1A	1A	1A	1A	1A	1A	1A	1A	1B
	°C	100	100	100	100	100	100	100	100	100	100	100	100
	Hours	2	2	2	2	2	2	2	2	2	2	2	2
D381	Existent Gum	g/100ml	1	1	3	1	23	15	2	3	19	42	17
D5006	Fuel System Icing Inhibitor Content	vol %	0.16	0.16	0.15	0.15	0.13	0.16	0.16	0.16	0.17	0.16	0.17
SWRI	Hy-LITE	RLU/1L	37	7	180	9	23	9	39	15	16	12	17
D5001	BOCLE	mm	0.51	0.51	0.49	0.51	0.48	0.47	0.52	0.54	0.48	0.45	0.48

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**Table C-5. HEFA SPK Blend Data – End of Test (EOT)**

Additives			HRJ-1	HRJ-2	HRJ-3	HRJ-4	HRJ-5	HRJ-6	HRJ-7	HRJ-8	HRJ-9	HRJ-10	HRJ-11
Biocide - Kathon FP 1.5			-	100 ppm	-	-	-	-	-	-	100 ppm	100 ppm	100 ppm
CN #1 - 2 ethyl hexyl nitrate (2EHN)			-	-	0.80 vol %	0.40 vol %	-	-	-	-	0.80 vol %	-	-
CN #2 - 2EHN, with storage stability additives			-	-	-	-	0.50 vol %	0.25 vol %	-	-	-	0.50 vol %	-
CN #3 - Di Tert Butyl Peroxide (DTBP)			-	-	-	-	-	-	0.50 vol %	0.25 vol %	-	-	0.50 vol %
SwRI Sample ID			CL12-3453	CL12-3454	CL12-3455	CL12-3456	CL12-3457	CL12-3458	CL12-3459	CL12-3460	CL12-3461	CL12-3462	CL12-3463
Description			HRJ-1, CAF-7815, 2/15/12	HRJ-2, CAF-7815, 2/15/12	HRJ-3, CAF-7815, 2/15/12	HRJ-4, CAF-7815, 2/15/12	HRJ-5, CAF-7815, 2/15/12	HRJ-6, CAF-7815, 2/15/12	HRJ-7, CAF-7815, 2/15/12	HRJ-8, CAF-7815, 2/15/12	HRJ-9, CAF-7815, 2/15/12	HRJ-10, CAF-7815, 2/15/12	HRJ-11, CAF-7815, 2/15/12
D1319	Aromatic Content												
	Aromatics	%	0.50	0.30	0.60	0.30	0.30	0.30	0.40	0.30	0.60	0.50	0.50
	Olefins	%	0.60	0.20	0.30	0.80	0.20	0.20	0.50	0.20	0.30	0.70	0.40
	Saturates	%	98.90	98.90	99.10	98.90	99.50	99.50	99.10	99.50	99.10	98.80	99.10
D2624	Electrical Conductivity	pS/m	119	78	56	75	2470	1239	59	92	78	2190	70
		°C	19.5	20.1	20.0	20.0	20.5	19.6	20.3	19.6	19.5	20.0	19.7
D3241	Jet Fuel Thermal Stability												
	Test Temperature	°C	275	275	275	275	275	275	275	275	275	275	275
	ASTM Code		<1	1	1	<1	1P	2	<1	<1	<1	1P	<1
	Maximum mm Hg	mm Hg	0.1	0.1	0	0	0.1	0	0	0	0	0.1	0.1
D3242	Acidity (Aviation)	mg KOH/g	0.004	0.006	0.008	0.007	0.007	0.013	0.012	0.015	0.008	0.010	0.011
D3701	Hydrogen by Nuclear Magnetic Resonance	mass %	15.40	15.19	15.18	15.44	15.43	15.49	15.29	15.27	15.35	15.26	15.31
D4052	Density at 15°C	g/mL	0.7616	0.7617	0.7632	0.7625	0.7627	0.7622	0.7620	0.7618	0.7634	0.7628	0.7620
D445	Viscosity - Kinematic at -20°C	cSt	5.90	5.87	5.85	5.88	5.88	5.89	5.87	5.89	5.87	5.87	5.90
D4809	Heat of Combustion – Net	MJ/kg	43.9	43.9	43.4	43.7	43.9	43.8	44.0	43.5	43.5	43.5	43.6
D5291	Carbon/Hydrogen												
	Carbon	mass %	84.86	84.72	84.73	84.76	84.41	84.75	84.42	84.98	84.82	84.59	84.63
	Hydrogen	mass %	15.37	15.31	15.30	15.25	15.20	15.23	15.19	15.30	15.26	15.24	15.28
	Carbon + Hydrogen	mass %	100.23	100.03	100.03	100.01	99.61	99.98	99.61	100.28	100.08	99.83	99.91
D5453	Sulfur – UV	ppm	0.8	0.9	2.5	1.7	1.6	1.1	0.4	0.8	2.4	1.9	0.6
D6890	IQT												
	Derived Cetane Number		59.48	59.89	90.73	82.25	83.24	77.55	73.05	68.59	90.36	83.44	72.58
	Ignition Delay	ms	3.392	3.366	2.457	2.616	2.595	2.724	2.846	2.987	2.463	2.591	2.859
D3948	MSEP		81	92	78	72	0	0	97	89	71	0	75
D86	Distillation												
	Initial Boiling Point	°C	149.9	150.0	149.9	150.5	146.7	150.5	148.9	149.9	151.7	148.6	148.7
	5% recovery	°C	161.9	162.4	161.3	162.4	162.1	162.9	161.1	161.6	163.2	162.7	161.8
	10% recovery	°C	166.5	167.1	166.2	167.0	165.9	166.4	164.6	166.4	166.2	166.5	165.7
	15% recovery	°C	172.4	172.5	171.1	172.1	171.8	172.3	172.2	171.3	172.4	172.0	172.1
	20% recovery	°C	178.1	178.6	177.5	178.4	177.4	177.8	178.0	178.5	177.6	177.9	178.5
	30% recovery	°C	192.2	192.4	191.1	192.9	192.6	192.6	192.9	193.2	191.8	192.7	193.0
	40% recovery	°C	208.2	209.3	208.1	209.7	209.9	209.4	209.2	209.9	209.2	210.0	209.6
	50% recovery	°C	225.9	226.2	225.8	227.1	226.7	226.8	226.3	226.6	226.7	226.8	226.2
	60% recovery	°C	241.3	241.6	241.6	242.3	242.5	242.4	242.0	242.4	242.6	242.2	241.9
	70% recovery	°C	255.1	255.4	255.4	256.0	256.3	255.8	255.6	255.7	255.9	256.4	255.9
	80% recovery	°C	266.5	266.8	266.8	267.1	267.3	266.9	266.8	266.9	267.1	267.3	266.7
	90% recovery	°C	274.6	274.7	274.9	275.1	275.2	274.9	274.8	274.8	274.8	275.1	274.8
	95% recovery	°C	278.3	278.5	278.8	279.1	278.9	278.6	278.7	278.5	278.8	278.8	278.7
	Final Boiling Point	°C	281.8	281.7	281.7	281.9	281.5	281.3	283.8	283.0	282.3	281.6	284.0
	Residue	mL	1.2	1.4	1.5	1.5	1.5	1.4	1.5	1.4	1.5	1.5	1.4
	Loss	mL	0.6	0.5	0.4	0.8	0.7	0.3	0.1	0.3	0.3	0.5	0.3
D5972	Freeze Point	°C	-55.3	-59.7	-55.8	-55.8	-57.6	-54.8	-55.8	-57.6	-58	-55.2	-54.8
D93	Flash Point (Pensky-Martin)	°C	45.5	45.5	45.5	44.5	45.5	44.5	41.5	45.5	45.5	44.5	44.5
D156	Saybolt color		19	19	19	19	13	15	15	15	17	11	15
D4294	Sulfur-Energy Dispersive	wt. %	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
D3227	Sulfur-Mercaptan	mass %	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003	<0.0003
D3338	Specific Energy	MJ/Kg	44.104	44.113	44.087	44.104	44.1	44.159	44.105	44.112	44.086	44.096	44.105
D1322	Smoke Point	mm	50	50	50	50	50	49	48	49	50	48	48
D976	Cetane Index		70.8	70.8	70	70.8	70.5	70.8	70.7	70.9	70.2	70.5	70.7
D130	Copper Strip Corrosion												
	Code		1B	1A	1B	1B	1A	1A	1A	1B	1A	1A	1A
	°C		100	100	100	100	100	100	100	100	100	100	100
	Hours		2	2	2	2	2	2	2	2	2	2	2
D381	Existent Gum	g/100ml	5.00	<0.5 mg/100 L	3.00	1.00	21.00	13.00	<0.5 mg/100 L	1.00	<0.5 mg/100 L	23.00	2.00
D5006	Fuel System Icing Inhibitor Content	vol %	0	0	0	0	0	0	0	0	0	0	0
SWRI	Hy-LITE (fuel)	RLU/1L	12	21	14	11	7	9	15	11	26	8	7
SWRI	Hy-LITE (water)	RLU/1L	20	10	13	20	32	7	36	17	24	17	7
D5001	BOCLE	mm	0.54	0.60	0.54	0.54	0.58	0.60	0.57	0.59	0.58	0.66	0.59

**Table C-6. B20 Blend Data – Start of Test (SOT)**

	<b>Additives</b>		<b>B20-1</b>	<b>B20-2</b>	<b>B20-3</b>	<b>B20-4</b>	<b>B20-5</b>	<b>B20-6</b>	<b>B20-7</b>	<b>B20-8</b>
	Biocide - Kathon FP 1.5		-	100 ppm	-	-	-	100 ppm	100 ppm	100 ppm
	CN #1 - 2 ethyl hexyl nitrate (2EHN)		-	-	0.80 vol %	-	-	0.80 vol %	-	-
	CN #2 - 2EHN, with storage stability additives		-	-	-	0.50 vol %	-	-	0.50 vol %	-
	CN #3 - Di Tert Butyl Peroxide (DTBP)		-	-	-	-	0.50 vol %	-	-	0.50 vol %
	<b>SwRI Sample ID</b>		<b>CL12-3475</b>	<b>CL12-3476</b>	<b>CL12-3477</b>	<b>CL12-3478</b>	<b>CL12-3479</b>	<b>CL12-3480</b>	<b>CL12-3481</b>	<b>CL12-3482</b>
	<b>Description</b>		<b>B20-1</b>	<b>B20-2</b>	<b>B20-3</b>	<b>B20-4</b>	<b>B20-5</b>	<b>B20-6</b>	<b>B20-7</b>	<b>B20-8</b>
<b>D1319</b>	<b>Aromatic Content</b>									
	Aromatics	%	See Report	See Report	See Report	See Report	See Report	See Report	See Report	See Report
	Olefins	%								
	Saturates	%								
<b>D2624</b>	<b>Electrical Conductivity</b>	pS/m	24	20	26	454	25	21	21	21
		°C	23.8	23.6	23.5	23.3	23.2	23.4	23.3	23.3
<b>D4052</b>	<b>Density at 15°C</b>	g/mL	0.8326	0.8327	0.8337	0.8333	0.8325	0.8337	0.8333	0.8325
<b>D445</b>	<b>Viscosity - Kinematic at 40°C</b>	cSt	2.02	2.03	2.00	2.05	2.01	2.02	2.02	2.04
<b>D5453</b>	<b>Sulfur – UV</b>	ppm	4.3	5.0	6.8	6.2	5.2	7.8	7.2	5.2
<b>D6890</b>	<b>IQT</b>									
	Derived Cetane Number		50.82	50.73	63.97	60.18	56.08	64.52	60.30	55.59
	Ignition Delay	ms	4.03	4.03	3.14	3.35	3.62	3.11	3.34	3.65
<b>D86</b>	<b>Distillation</b>									
	Initial Boiling Point	°C	174.3	175.3	166.1	170.5	168.5	167.5	170.7	169.4
	5% recovery	°C	192.9	192.9	190.7	191.7	191.2	190.6	191.1	192
	10% recovery	°C	198.7	198.3	196.5	197.5	198.5	195.8	196.7	198.5
	15% recovery	°C	203.5	204	203.5	203.4	203.9	203	203.2	203.9
	20% recovery	°C	208.6	208.9	208.5	208	209.2	207.5	207.9	208.5
	30% recovery	°C	218	218.7	218.3	218.1	218.3	218	217.6	218.1
	40% recovery	°C	228.2	228.2	228.2	228.2	228.1	227.9	227.9	228
	50% recovery	°C	239.8	240.1	240.1	239.5	240.1	240.1	239.4	239.8
	60% recovery	°C	256.2	256.1	256.1	255.7	256.1	256.1	255.7	256.2
	70% recovery	°C	280.9	280.2	279.8	279.5	279.9	280.1	279.4	279.7
	80% recovery	°C	315.6	315	314.1	314	313.9	314.6	313.2	314
	90% recovery	°C	334.4	334.4	335	334.7	334.8	335	334.4	347.7
	95% recovery	°C	339.6	339.4	340.9	340.2	341.3	341.4	339.9	341.3
	Final Boiling Point	°C	344.7	345	347.5	346.3	347.3	346.5	346.7	347.8
	Residue	mL	0.8	0.9	1.0	1.0	1.0	1.0	0.9	0.9
	Loss	mL	0.4	0.3	0.2	0.1	0.3	0.5	0.2	0.3
	T90-T10	°C	135.7	136.1	138.5	137.2	136.3	139.2	137.7	149.2
<b>D93</b>	<b>Flash Point (Pensky-Martin)</b>	°C	59.5	60.0	62.0	62.0	58.5	57.5	57.5	56.5
<b>D976</b>	<b>Cetane Index</b>		46.2	46.3	45.9	45.9	46.4	45.9	45.9	46.3
<b>D130</b>	<b>Copper Strip Corrosion</b>									
	Code		1A	1A	1A	1A	1A	1A	1A	1A
	°C		50	50	50	50	50	50	50	50
	Hours		3	3	3	3	3	3	3	3
<b>SWRI</b>	<b>Hy-LITE</b>	RLU/1L	36	13	8	12	14	17	8	25
<b>D6079</b>	<b>HFRR</b>									
	Major Axis	mm	0.23	0.29	0.27	0.26	0.24	0.24	0.23	0.25
	Minor Axis	mm	0.16	0.23	0.22	0.22	0.16	0.16	0.17	0.15
	Wear Scar Diameter	mm	0.193	0.261	0.245	0.243	0.199	0.204	0.197	0.200
	Temperature	°C	60	60	60	60	60	60	60	60
<b>D6371</b>	<b>CFPP</b>	°C	-23	-23	-23	-23	-23	-23	-23	-23
<b>EN15751</b>	<b>Rancimat</b>	Hours	10.6	9.6	8.5	14.2	10.1	6.7	12.2	8.5
<b>D524</b>	<b>10% Bottom</b>	wt %	0.04	0.01	0.12	0.05	0.06	0.13	0.09	0.02
<b>D2500</b>	<b>Cloud Point</b>	°C	-13	-14	-13	-13	-13	-15	-15	-13
<b>D482</b>	<b>Ash</b>									
	Ash	mass %	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Mass of Sample taken	g	50.1196	50.0715	50.1924	50.0733	50.0266	51.881	50.0237	50.6195
<b>D664</b>	<b>Acid Number</b>									
	Acid No. Inflection	mg KOH/g	-	-	0.08	-	-	-	-	-
	Acid No. Buffer	mg KOH/g	<0.05	<0.05	0.05	<0.05	<0.05	<0.05	<0.05	<0.05
<b>EN 14078</b>	<b>FAME Content</b>	vol %	24	24	23.7	23.8	24	23.7	23.8	23.5
	Note:		10vol% dilution w/cyclohexane.	10vol% dilution w/cyclohexane.	10vol% dilution w/cyclohexane.	10vol% dilution w/cyclohexane.	10vol% dilution w/cyclohexane.	10vol% dilution w/cyclohexane.	10vol% dilution w/cyclohexane.	10vol% dilution w/cyclohexane.

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**Table C-7. B20 Blend Data – End of Test (EOT)**

	Additives		B20-1	B20-2	B20-3	B20-4	B20-5	B20-6	B20-7	B20-8
			-	100 ppm	-	-	-	100 ppm	100 ppm	100 ppm
	Biocide - Kathon FP 1.5		-	-	-	-	-	-	-	-
	CN #1 - 2 ethyl hexyl nitrate (2EHN)		-	-	0.80 vol %	-	-	0.80 vol %	-	-
	CN #2 - 2EHN, with storage stability additives		-	-	-	0.50 vol %	-	-	0.50 vol %	-
	CN #3 - Di Tert Butyl Peroxide (DTBP)		-	-	-	-	0.50 vol %	-	-	0.50 vol %
	SwRI Sample ID		CL12-3475	CL12-3476	CL12-3477	CL12-3478	CL12-3479	CL12-3480	CL12-3481	CL12-3482
	Description		B20-1	B20-2	B20-3	B20-4	B20-5	B20-6	B20-7	B20-8
D1319	Aromatic Content									
	Aromatics	%	32.2	36.9	36.4	35.6	34.8	36.6	36.9	33.8
	Olefins	%	0.8	1.0	0.6	1.0	1.2	0.9	1.3	1.1
	Saturates	%	67	62.1	63	63.4	64	62.5	61.8	65.1
D2624	Electrical Conductivity	pS/m	88	89	189	4690	167	203	3980	136
		°C	20.0	18.9	20.1	20.0	19.5	19.2	19.9	18.9
D4052	Density at 15°C	g/mL	0.8342	0.8337	0.8340	0.8336	0.8328	0.8340	0.8336	0.8336
D445	Viscosity - Kinematic at 40°C	cSt	2.09	2.10	2.03	2.05	2.02	2.03	2.05	2.10
D5453	Sulfur – UV	ppm	5.1	5.6	6.9	6.6	4.7	7.9	6.5	5.2
D6890	IQT									
	Derived Cetane Number		59.71	57.96	64.33	60.82	57.00	64.43	60.62	59.61
	Ignition Delay	ms	3.377	3.488	3.117	3.311	3.552	3.112	3.323	3.383
D86	Distillation									
	Initial Boiling Point	°C	167.4	170.1	164.1	160.4	166.7	164.9	163	165.2
	5% recovery	°C	191.4	192.1	190.2	190.2	190.1	189.9	190.2	189.3
	10% recovery	°C	196.5	197.4	194.2	195.9	196.7	194.4	196.3	197.7
	15% recovery	°C	202.7	203.7	202.2	203	202.1	202	202.8	203.2
	20% recovery	°C	207.5	208.6	207.4	207.5	207.8	207.3	207.9	208.3
	30% recovery	°C	217.3	217.7	217.6	217.9	217.2	217.1	217.3	217.3
	40% recovery	°C	227.1	228.2	227.8	227.8	227.5	226.8	227.5	227.1
	50% recovery	°C	238.7	239.9	239	239.8	239.2	238.4	239.3	238.7
	60% recovery	°C	254.3	255	255.2	255.3	255	254.1	254.2	254.3
	70% recovery	°C	278	279.2	279.5	279.9	279.1	277.8	279.8	278
	80% recovery	°C	312.3	313.5	314.3	315.2	313.1	312	314.1	312.8
	90% recovery	°C	334.7	334.4	334.9	334.4	334.1	334.4	334.8	334.8
	95% recovery	°C	340.9	341.2	340.9	341.1	340.6	340.2	341	341.9
	Final Boiling Point	°C	342.2	343.4	343.7	346.2	344.1	344.8	347	342.9
	Residue	mL	0.8	1	1.0	1.0	1.0	0.9	1	1
	Loss	mL	0.1	0.2	0.3	0.6	0.1	0.1	0.4	0.2
D93	Flash Point (Pensky-Martin)	°C	59.5	60.5	57.5	60.5	57.5	58.5	61.5	57.5
D976	Cetane Index		45.4	45.9	45.5	45.9	46	45.3	45.7	45.6
D130	Copper Strip Corrosion									
	Copper Corrosion		1A	1A	1A	1A	1A	1A	1A	1A
	Temperature	°C	50	50	50	50	50	50	50	50
	Hours	Hours	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
SWRI	Hy-LITE (fuel)		13	17	21	150	23	21	11	15
SWRI	Hy-LITE (water)		21	22	15	92	31	11	13	83
D6079	HFRR									
	Major Axis	mm	0.41	0.4	0.27	0.22	0.38	0.3	0.27	0.4
	Minor Axis	mm	0.35	0.34	0.24	0.19	0.3	0.3	0.23	0.3
	Wear Scar Diameter	mm	0.38	0.37	0.26	0.2	0.34	0.3	0.25	0.350
	Temperature	°C	60	60	60	60	60	60	60	60
D6371	CFPP	°C	-22	-22	-23	-22	-22	-22	-22	-22
EN15751	Rancimat	Hours	0.5	0.9	3.1	5.4	3.2	3.2	4.4	0.9
D524	10% Bottom		0.08	0.07	0.13	0.09	0.09	0.02	0.07	0.13
D2500	Cloud Point		-10	-10	-11	-10	-12	-10	-12	-10
D482	Ash									
	Ash		<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Mass of Sample taken		50.0087	50.0241	50.0077	50.0125	50.0139	50.0361	50.0331	50.0335
D664	Acid Number									
	Acid No. Inflection		N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	Acid No. Buffer		0.13	0.09	0.05	0.08	0.08	0.06	0.11	0.13
EN 14078	FAME Content		24.9	25.5	25.1	25.2	25.4	25.1	25.1	25.4
	Note:		10vol% diluton w/cyclohexane.	10vol% diluton w/cyclohexane.	10vol% diluton w/cyclohexane.	10vol% diluton w/cyclohexane.	10vol% diluton w/cyclohexane.	10vol% diluton w/cyclohexane.	10vol% diluton w/cyclohexane.	10vol% diluton w/cyclohexane.

**APPENDIX D**  
**Statistical Analysis of Test Data**

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## Statistical Evaluation of Data Generated in Test Program

In evaluating the results of the test program, one of the ways to inspect the data is to compare it to the expected precision for the method involved. For most of the tests in this program there is an established expectation of precision. There are also some common estimation methods that can be applied. For each test with established precision, an analysis was prepared and placed in the table for the type of material being studied. Since there are two basic types of material in this program, there are two tables:

- 1) Table D-1. Data Analysis Precision Review for Jet Fuel Type Samples
- 2) Table D-2. Data Analysis Example for B20 Type Samples

Note that each of these tables starts with a guide to how the data is arranged.

First, consider what is meant by the term “precision”. Here is the ASTM definition:

precision,  $n$ —the closeness of agreement between independent test results obtained under stipulated conditions.

In tests with numerical results, it is unlikely that any two, or more, tests will have identical results. Evaluating the precision of a method provides a benchmark against which to judge if the difference in results is significant. Basically, data within the established precision should be considered equivalent. Data outside of the established precision should be inspected to determine if the results show a real difference.

Basically, there are two generally reported types of precision, repeatability,  $r$ , and reproducibility,  $R$ . Repeatability is the ability of the single operator in a laboratory, using the same sample, to get equivalent results for a test method. Reproducibility is the ability of different operators in separate laboratories, using a different aliquot of the same sample, to get equivalent results for a test method. These values are established for a 95% confidence interval, which means that no more than one test in twenty (for the same sample) should fall outside of the expected precision. Note: these values are sometimes confused with a standard deviation and thought to refer to a  $\pm$  range. This is not true; the precision value establishes the maximum range within which the maximum and minimum values should be found. (Example: The

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reproducibility for a series of Pensky-Martens flash points, ASTM D93, averaging 42 °C would be 3 °C. That does not mean values of  $42 \pm 3$  °C are to be considered equivalent, it means that the highest and lowest values should not be farther apart than 3 °C.)

In practice, the effort being conducted here is not strictly repeatability, as the presence of the additives makes each sample different. This would fall into a category referred to as intermediate precision, the closeness of agreement between test results obtained under specified intermediate precision conditions. In this case the intermediate conditions are identical base materials with different additives. So the question becomes “Does the presence of the additives significantly impact the results so that the given precision values for repeatability and/or reproducibility no longer constrains the system?”

To make this comparison, one approach would be to simply calculate the difference between the minimum and maximum values for a given property and then compare that value to the expected precision. That would be a direct analysis but it could be adversely affected by the one-in-twenty test that is anticipated by the 95% confidence interval. Rather, for this exercise the analysis was made by comparing the standard deviation of the values generated for a specific test performed on a specific fuel type, with and without additives. The manner in which it was calculated reduces the affect of a single test. However, as noted above, precision is not the same as standard deviation, so how can they be compared? The calculated precision is equal to 2.8 times the weighted (square root of average summed squares) standard deviation of the property in question. The intermediate precision of this exercise can be approximated by multiplying the standard deviation of the test results by 2.8.

This calculated intermediate precision was compared to the repeatability and reproducibility values quoted in the respective method. Doing so resulted in an excessive amount of the tests being reported as problematic. A review of information from the ASTM Inter Laboratory Crosscheck Program (ILCP) showed that for real world testing, on the same sample, it was not unusual for the reproducibility of the ILCP tests to be as much as twice that quoted in the method. Based on that observation, the evaluation criterion was been changed to compare the

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estimated test precision to two times the method reproducibility, 2R. No adjustment was made to the repeatability, r, value. This comparison is annotated by color coding the result as noted in analysis tables.

The concept is that subtle effects, unlike the effects on derived cetane number (DCN) or thermal stability (JFTOT), may be best identified by the effect on test statistics. This is, by its nature, the more difficult part of the data analysis. It is likely that any effects noted will be classified as 'possible' at best. Still, this analysis will be valuable in developing a listing of potential effects of any routine use of the additives being tested.



**Table D-1. Data Analysis Precision Review for Jet Fuel Type Samples**

**How Each Test is Analyzed**

Test	FT SPK			HEFA SPK			Method Repeatability		Method Reproducibility		Method Calculation
	Average	St. Dev.	~r/R	Average	St. Dev.	~r/R					
<b>Start of Test</b>											
Report Item, Units	A <sub>FT</sub>	B <sub>FT</sub>	C <sub>FT</sub>	A <sub>HEFA</sub>	B <sub>HEFA</sub>	C <sub>HEFA</sub>	r <sub>FT</sub>	r <sub>HEFA</sub>	R <sub>FT</sub>	R <sub>HEFA</sub>	r = Formula or Table Value R = Formula or Table Value
<b>End of Test</b>											

**Definition of Terms**

Test	ASTM number of the test being consider
Start of Test	Values for the test at the start of the program
End of Test	Values for the test at the end of the program
Report Item, Units	What is being reported and the units, if appropriate
Average (A)	Average value for the samples
St. Dev. (B)	Standard deviation for the samples
~r/R (C)	Precision approximation = 2.8 x Std. Dev.
Method Repeatability, r	Repeatability expected for the value in the test program
Method Reproducibility, R	Reproducibility expected for the value in the test program
Method Calculation	Source of the precision information for the method

**Evaluation of Test Precision Approximation**

C < r	Test precision approximation, C, is less than the method repeatability, r, for the average value, A.
r < C < 2R	Test precision approximation, C, is less than two times* the method reproducibility, R, for the average value, A, but greater than the method repeatability
C > 2R	Test precision approximation, C, is greater than two times the method reproducibility, R, for the average value, A.

\*In ASTM Inter Laboratory Crosscheck Programs (ILCP) it is not unusual for tests to have R values much higher than those generated in the method precision study

D1319	FT SPK			HEFA SPK			Method Repeatability		Method Reproducibility		Method Calculation
	Average	St. Dev.	~r/R	Average	St. Dev.	~r/R					
<b>Start of Test</b>											
Aromatics, vol %	1.35	0.35	0.98	0.44	0.14	0.38	0.70	0.70	1.50	1.50	Table 3 from Standard
Olefins, vol %	0.59	0.14	0.39	0.43	0.14	0.40	0.40	0.40	1.70	1.70	
Saturates, vol %	98.05	0.42	1.18	99.14	0.25	0.71	0.30	0.30	2.40	2.40	
<b>End of Test</b>											
Aromatics, vol %	1.05	0.35	0.98	0.42	0.13	0.35	0.70	0.70	1.50	1.50	
Olefins, vol %	0.41	0.13	0.36	0.40	0.22	0.61	0.40	0.40	1.70	1.70	
Saturates, vol %	98.55	0.39	1.09	99.13	0.26	0.73	0.30	0.30	2.40	2.40	
D2624	FT SPK			HEFA SPK			Method Repeatability		Method Reproducibility		Method Calculation
	Average	St. Dev.	~r/R	Average	St. Dev.	~r/R					
<b>Start of Test</b>											
Conductivity, pS/m	128	210	588	70	117	328	22	15	21	13	Extrapolate from Table 1
<b>End of Test</b>											
Conductivity, pS/m	728	1489	4168	593	928	2598	63	56	95	80	Extrapolate from Table 1

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**Table D-1. Data Analysis Precision Review for Jet Fuel Type Samples (Cont.)**

	FT SPK			HEFA SPK			Method Repeatability		Method Reproducibility		Method Calculation
	Average	St. Dev.	~r/R	Average	St. Dev.	~r/R					
<b>D3242</b>											
Start of Test											
Acid Number, mgKOH/l	0.016	0.0034	0.0094	0.016	0.0047	0.0133	0.0017	0.0017	0.0052	0.0052	$r = 0.0132 \times \sqrt{\text{average}}$ $R = 0.0406 \times \sqrt{\text{average}}$
End of Test											
Acid Number, mgKOH/l	0.008	0.002	0.0046	0.009	0.003	0.0093	0.0012	0.0013	0.0037	0.0039	
<b>D3701</b>											
Start of Test											
Hydrogen, mass %	15.41	0.07	0.20	15.25	0.06	0.18	0.09	0.09	0.11	0.11	$r = \text{from para 13.1.1 of method}$ $R = \text{from para 13.1.2 of method}$
End of Test											
Hydrogen, mass %	15.45	0.12	0.34	15.33	0.10	0.29	0.09	0.09	0.11	0.11	
<b>D4052</b>											
Start of Test											
Density	0.7425	0.0020	0.0056	0.7626	0.0006	0.0017	0.00045	0.00045	0.0022	0.0015	$r = \text{Table 2 of the Method}$
End of Test											
Density	0.7423	0.0019	0.0054	0.7624	0.0006	0.0017	0.00045	0.00045	0.0022	0.0015	$R = 0.00190 - 0.0344 (D - 0.75)$
<b>D445</b>											
Start of Test											
Viscosity, mm <sup>2</sup> /sec	2.569	0.036	0.101	5.829	0.045	0.126	0.018	0.041	0.049	0.111	$r = 0.007 \times$ $R = 0.019 \times$
End of Test											
Viscosity, mm <sup>2</sup> /sec	2.585	0.033	0.091	5.879	0.015	0.042	0.018	0.041	0.049	0.112	
<b>D4809</b>											
Start of Test											
MI/kg	43.70	0.15	0.407	43.63	0.15	0.409	0.099	0.099	0.234	0.234	$r = \text{from para 13.1.1 of method}$ $R = \text{from para 13.1.2 of method}$
End of Test											
MI/kg	43.97	0.238	0.665	43.70	0.213	0.596	0.099	0.099	0.234	0.234	
<b>D5291</b>											
Start of Test											
Carbon	84.42	0.17	0.48	84.46	0.10	0.28	0.96	0.96	2.39	2.39	$r_c = (x + 48.48)0.0072$
Hydrogen	15.42	0.08	0.23	15.28	0.10	0.29	0.46	0.45	0.91	0.90	$R_c = (x + 48.48)0.0018$
Carbon + Hydrogen	99.84	0.21		99.74	0.06						$r_H = (x^{0.5})0.1162$ $R_H = (x^{0.5})0.2314$
End of Test											
Carbon	84.56	0.18	0.50	84.70	0.17	0.49	0.96	0.96	2.39	2.40	
Hydrogen	15.35	0.30	0.84	15.27	0.05	0.15	0.46	0.45	0.91	0.90	
Carbon + Hydrogen	99.91	0.25		99.96	0.22						
<b>D5453</b>											
Start of Test											
Sulfur, mg/kg	2.09	0.69	1.94	1.67	0.78	2.20	0.31	0.26	1.01	0.85	$r = 0.1788 \times^{0.75}$ $R = 0.5797 \times^{0.75}$ Table 3 of D5453
End of Test											
Sulfur, mg/kg	1.90	0.77	2.15	1.34	0.72	2.03	0.29	0.22	0.94	0.72	
<b>D6890</b>											
Start of Test											
Derived Cetane Number	76.97	11.59	32.45	78.32	11.40	31.92	0.76	0.76	5.85	5.99	$r_{DCN} = 0.76$
Ignition Delay	2.80	0.35	0.98	2.76	0.31	0.88	0.017	0.015	0.158	0.155	$r_{ID} = 0.0465 \times (ID - 2.432)$ $R_{DCN} = 0.0987 \times (DCN - 17.67)$ $R_{ID} = 0.0777 \times (ID - 0.7679)$
End of Test											
Derived Cetane Number	73.51	11.17	31.29	76.47	10.85	30.39	0.76	0.76	5.51	5.80	
Ignition Delay	2.90	0.37	1.05	2.81	0.33	0.91	0.022	0.018	0.166	0.159	

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**Table D-1. Data Analysis Precision Review for Jet Fuel Type Samples (Cont.)**

	FT SPK			HEFA SPK			Method Repeatability		Method Reproducibility		Method Calculation
	Average	St. Dev.	~r/R	Average	St. Dev.	~r/R					
<b>D3948</b>											
Start of Test											
MSEP #	80	24	68	57	37	103	15	24	17	27	From Fig. 10, D3948
End of Test	68	44	124	60	39	109	18	23	22	26	
<b>D86</b>											
Start of Test											
10% recovery, °C	163.0	0.4	1.06	166.0	0.7	2.06	1.53	1.56	3.59	3.65	$r_{10} = 0.0094T$ $R_{10} = 0.022T$ $r_{50} = 0.94$ $R_{50} = 2.97$ $r_{90} = 0.0041T$ $R_{90} = 0.015T$ $r_{FBP} = 2.2$ $R_{FBP} = 7.1$
50% recovery, °C	170.9	0.6	1.72	226.7	0.7	1.91	0.94	0.94	2.97	2.97	
90% recovery, °C	190.1	1.7	4.84	275.2	0.3	0.75	0.78	1.13	2.85	4.13	
Final Boiling Point, °C	215.3	3.6	10.19	282.4	0.9	2.54	2.2	2.2	7.1	7.1	
End of Test											
10% recovery, °C	162.8	0.4	1.03	166.2	0.7	1.90	1.53	1.56	3.58	3.66	
50% recovery, °C	170.7	0.6	1.60	226.5	0.4	1.15	0.94	0.94	2.97	2.97	
90% recovery, °C	189.5	1.7	4.65	274.9	0.2	0.51	0.78	1.13	2.84	4.12	
Final Boiling Point, °C	214.1	4.0	11.32	282.2	0.9	2.63	2.2	2.2	7.1	7.1	
<b>D5972</b>											
Start of Test											
Freeze Point, °C	-54.52	0.51	1.42	-54.82	1.04	2.92	0.54	0.54	0.80	0.80	Section 12 of Method
End of Test	-55.77	1.31	3.68	44.86	1.21	3.38	0.54	0.54	0.80	0.80	
<b>D93</b>											
Start of Test											
Flash Point, °C	42.6	0.50	1.41	43.0	1.48	4.15	1.24	1.25	3.03	3.05	$r = 0.029X$ $R = 0.071X$
End of Test	42.23	0.93	2.61	44.86	1.21	3.38	1.22	1.30	3.00	3.19	
<b>D156</b>											
Start of Test											
Saybolt Color	25.55	2.50	7.01	19.55	1.13	3.16	1	1	2	2	From Section 13 of Method
End of Test	20.91	3.53	9.90	16.09	2.74	7.66	1	1	2	2	
<b>D3338</b>											
Start of Test											
Specific Heat, MJ/kg	44.15	0.03	0.083	44.10	0.01	0.033	0.021	0.021	0.046	0.046	$r = \text{from para 9.1.1 of method}$ $R = \text{from para 9.1.2 of method}$
End of Test	44.16	0.030	0.083	44.11	0.019	0.055	0.021	0.021	0.046	0.046	
<b>D1322</b>											
Start of Test											
Smoke Point, mm	24.32	0.93	2.60	24.82	1.01	2.82	2.76	2.79	3.77	3.82	Manual $r = 0.06840 (x + 16)$ $R = 0.09363 (x + 16)$ Automatic $r = 0.02231x$ $R = 0.1651 (x + 30)$
End of Test	46.91	1.30	3.64	49.27	0.90	2.53	4.30	4.46	5.89	6.11	
							1.05	1.10	1.27	1.31	

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**Table D-1. Data Analysis Precision Review for Jet Fuel Type Samples (Cont.)**

	FT SPK			HEFA SPK			Method Repeatability	Method Reproducibility	Method Calculation
	Average	St. Dev.	~r/R	Average	St. Dev.	~r/R			
<b>D976</b>									
Start of Test									
Cetane Index	56.00	0.67	1.87	70.56	0.39	1.08	n/a	n/a	No Precision Stated in Method
End of Test									
Cetane Index	55.95	0.68	1.89	70.61	0.28	0.80	n/a	n/a	
<b>D381</b>									
Start of Test									
	22.45	9.11	25.52	11.55	13.14	36.79	6.18 3.46	9.21 5.49	r = 0.5882 + 0.2490x R = 2.941 + 0.2794x
End of Test									
	6.43	9.34	26.16	8.63	9.13	25.57	2.19 2.74	4.74 5.55	
<b>D5006</b>									
Start of Test									
FSII, vol %	0.159	0.033	0.092	0.157	0.011	0.031	0.009   0.009	0.021   0.021	r = from para 12.1.1 of method R = from para 12.1.2 of method
End of Test									
FSII, vol %	0.003	0.005	0.013	0.000	0.000	0.000	0.009   0.009	0.021   0.021	
<b>D5001</b>									
Start of Test									
WSD, mm	0.49	0.03	0.09	0.49	0.03	0.07	0.014 0.015	0.017 0.017	r = 0.08580 * x <sup>2.5083</sup> R = 0.09857 * x <sup>2.5083</sup>
End of Test									
WSD, mm	0.56	0.03	0.09	0.58	0.04	0.10	0.020 0.022	0.023 0.025	

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**Table D-2. Data Analysis Example for B20 Type Samples**

**How Each Test is Analyzed**

Test	FT SPK			Method Repeatability	Method Reproducibility	Method Calculation
	Average	St. Dev.	$\sim r/R$			
<b>Start of Test</b>						
Report Item, Units	A <sub>B20</sub>	B <sub>B20</sub>	C <sub>B20</sub>	r <sub>B20</sub>	R <sub>B20</sub>	r = Formula or Table Value R = Formula or Table Value
<b>End of Test</b>						

**Definition of Terms**

Test	ASTM number of the test being consider
Start of Test	Values for the test at the start of the program
End of Test	Values for the test at the end of the program
Report Item, Units	What is being reported and the units, if appropriate
Average (A)	Average value for the samples
St. Dev. (B)	Standard deviation for the samples
$\sim r/R$ (C)	Precision approximation = 2.8 x Std. Dev.
Method Repeatability, r	Repeatability expected for the value in the test program
Method Reproducibility, R	Reproducibility expected for the value in the test program
Method Calculation	Source of the precision information for the method

**Evaluation of Test Precision Approximation**

C < r	Test precision approximation, C, is less than the method repeatability, r, for the average value, A.
r < C < 2R	Test precision approximation, C, is less than two times* the method reproducibility, R, for the average value, A, but greater than the method repeatability
C > 2R	Test precision approximation, C, is greater than two times the method reproducibility, R, for the average value, A.

\*In ASTM Inter Laboratory Crosscheck Programs (ILCP) it is not unusual for tests to have R values much higher than those generated in the method precision study

D1319	B20			Method Repeatability	Method Reproducibility	Method Calculation
	Average	St. Dev.	$\sim r/R$			
<b>Start of Test</b>						
Aromatics, vol %						Table 3 from Standard
Olefins, vol %						
Saturates, vol %						
<b>End of Test</b>						
Aromatics, vol %	35.40	1.70	4.75	1.50	3.30	
Olefins, vol %	0.99	0.22	0.62	0.40	1.70	
Saturates, vol %	63.61	1.73	4.86	1.70	5.30	

D2624	B20			Method Repeatability	Method Reproducibility	Method Calculation
	Average	St. Dev.	$\sim r/R$			
<b>Start of Test</b>						
Conductivity, pS/m	77	153	427	16	14	Extrapolate from Table 1
<b>End of Test</b>						
Conductivity, pS/m	1193	1949	5458	85	145	Extrapolate from Table 1

D4052	B20			Method Repeatability	Method Reproducibility	Method Calculation
	Average	St. Dev.	$\sim r/R$			
<b>Start of Test</b>						
Density	0.8330	0.0005	0.0015	0.00016	0.00052	r = Table 2 of the Method
<b>End of Test</b>						
Density	0.8337	0.0004	0.0012	0.00016	0.00052	R = Table 4 of the Method

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**Table D-2. Data Analysis Example for B20 Type Samples (Cont.)**

	B20			Method Repeatability	Method Reproducibility	Method Calculation
	Average	St. Dev.	~r/R			
<b>D445</b>						
<b>Start of Test</b>						
Viscosity, mm <sup>2</sup> /sec	2.024	0.016	0.045	0.014	0.038	r = 0.007 x R = 0.019 x
<b>End of Test</b>						
Viscosity, mm <sup>2</sup> /sec	2.059	0.033	0.093	0.014	0.039	
<b>D5453</b>						
<b>Start of Test</b>						
Sulfur, mg/kg	5.96	1.23	3.43	0.68	2.21	r = 0.1788 X <sup>0.75</sup> R = 0.5797X <sup>0.75</sup> Table 3 of D5453
				0.6	1.9	
<b>End of Test</b>						
Sulfur, mg/kg	6.06	1.09	3.05	0.69	2.24	
<b>D6890</b>						
<b>Start of Test</b>						
Derived Cetane Num	57.77	5.37	15.03	0.76	3.96	r <sub>DCN</sub> = 0.76 r <sub>ID</sub> = 0.0465 x (ID - 2.432) R <sub>DCN</sub> = 0.0987 x (DCN - 17.67) R <sub>ID</sub> = 0.0777 x (ID - 0.7679)
Ignition Delay	3.53	0.36	1.02	0.051	0.215	
<b>End of Test</b>						
Derived Cetane Num	60.56	2.68	7.51	0.76	4.23	
Ignition Delay	3.33	0.16	0.44	0.042	0.199	
<b>D86</b>						
<b>Start of Test</b>						
10% recovery, °C	197.6	1.1	3.10	1.86	4.35	r <sub>10</sub> = 0.0094T R <sub>10</sub> = 0.022T r <sub>50</sub> = 0.94 R <sub>50</sub> = 2.97 r <sub>90</sub> = 0.0041T R <sub>90</sub> = 0.015T r <sub>F BP</sub> = 2.2 R <sub>F BP</sub> = 7.1
50% recovery, °C	239.9	0.3	0.81	0.94	2.97	
90% recovery, °C	336.3	4.6	12.92	1.38	5.04	
Final Boiling Point, °C	346.5	1.1	3.16	2.2	7.1	
<b>End of Test</b>						
10% recovery, °C	196.1	1.3	3.56	1.84	4.32	
50% recovery, °C	239.1	0.5	1.49	0.94	2.97	
90% recovery, °C	334.6	0.3	0.78	1.37	5.02	
Final Boiling Point, °C	344.3	1.6	4.58	2.2	7.1	
<b>D93</b>						
<b>Start of Test</b>						
Flash Point, °C	59.2	2.07	5.79	1.72	4.20	r = 0.029X R = 0.071X
<b>End of Test</b>						
Flash Point, °C	59.13	1.60	4.47	1.71	4.20	
<b>D976</b>						
<b>Start of Test</b>						
Cetane Index	46.1	0.22	0.62	n/a	n/a	No Precision Stated in Method
<b>End of Test</b>						
Cetane Index	45.7	0.26	0.72	n/a	n/a	
<b>D6079, HFRR @ 60°C</b>						
<b>Start of Test</b>						
WSD, mm	217.8	27	76	80	136	r = Para 15.1.1 of Method R = Para 15.1.2 of Method
<b>End of Test</b>						
WSD, mm	306.3	65	181	80	136	

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**Table D-2. Data Analysis Example for B20 Type Samples (Cont.)**

	B20			Method Repeatability	Method Reproducibility	Method Calculation
	Average	St. Dev.	~r/R			
<b>D6371</b>						
Start of Test CFPP, °C	-23.00	0.00	0.00	1.76	4.90	r = 1.76 R = 0.102*(25-X)
End of Test CFPP, °C	-22.13	0.35	0.99	1.76	4.81	
<b>EN15751</b>						
Start of Test Rancimat, hrs	10.05	2.34	6.56	0.36	2.29	r = 0.22027 + 0.04344x R = 0.37269 + 0.19038x
End of Test Rancimat, hrs	2.70	1.78	4.98	0.26	0.89	
<b>D524 Ramsbottom Carbon</b>						
Start of Test 10% Bottoms	0.065	0.044	0.124	0.024	0.031	Log r = 0.75238 log x + 0.23682 (log x) <sup>2</sup> - 1.06940 Log R = 0.78907 log x + 0.19014 (log x) <sup>2</sup> - 0.85333
End of Test 10% Bottoms	0.085	0.035	0.099	0.025	0.034	
<b>D2500</b>						
Start of Test Cloud Point, °C	-13.63	0.92	2.57	2.00	3	r = Method Para 10.3.1 R = Method Para 10.3.2
End of Test Cloud Point, °C	-10.63	0.92	2.57	2.00	3	
<b>D664</b>						
Start of Test Acid Number	only one sample registered any acid at all					r = Method Para 22.1.1 R estimated by ratio from B100 Precision
End of Test Acid Number	0.091	0.030	0.084	0.038	0.074 (est)	
<b>EN 14078</b>						
Start of Test FAME Content	23.81	0.18	0.51	0.58	1.85	r = 0.01505 (x + 14.905) R = 0.04770 (x + 14.905)
End of Test FAME Content	25.21	0.20	0.57	0.60	1.91	

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